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ADVANCEMENTS IN MATERIALS SCIENCE—FOREIGN (U)
Report No. 1
Reviews of Soviet Technologies

1. Figure 3-1 (page 3-5)—add following information to caption:
   1 (without coating), 2 (with Mo + Si coating), 3 (with Si coating).

2. Figure 3-2 (page 3-6)—add following information to caption:
   1 (without coating), 2 (with Ni + Al coating), 3 (with Si coating).

3. Figure 3-3 (page 3-7)—add following information to caption:
   1 (AN5 alloy), 2 (AN6a alloy), 3 (iodide titanium), 4 (AT10 alloy),
   5 (IVT1 alloy), 6 (AN5 alloy, aluminum coating), 7 (nicchrome alloy,
   80% Ni + 20% Cr), 8 (AN6 alloy).

4. Figure 3-4 (page 3-8)—add following information to caption:
   1 (IVT1 alloy), 2 (ST4 alloy), 3 (ST5 alloy), 4 (iodide titanium),
   5 (IVT1 alloy, aluminum coated, annealed at 800°C for 1 hour).

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ADVANCEMENTS IN MATERIALS SCIENCE—FOREIGN (U)

Report No. 1
Reviews of Soviet Technologies

J. Harold Mashburn, CPT, Cm1C

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PREFACE

This study is a collection of articles assessing significant Soviet achievements in materials science. It reviews Soviet efforts for relating the composition, structure, and processing of materials to their properties and uses. The topics selected for coverage include: key developments in Soviet metallurgy, fracture analysis of materials, research efforts in titanium coatings, composite materials developments, “glue-welding” technology, and developments in optical window materials.

Data for the reports were obtained from open-source scientific and technical literature dating from 1968. Because of the varying nature of the subjects covered, the articles are written as independent sections with a summary, discussion section, and bibliography.

This study and future editions are intended to provide an expedient means of presenting concise and current coverage for a broad range of materials topics. They complement current materials studies by broadening the scope of coverage and by addressing the technical areas which are narrowly covered in the basic material and system studies. Technical details are presented in conjunction with related processing and production technologies.

This study is directed to Department of Defense materials planners and research personnel concerned with development of advanced materials, materials theory, and processing technology.

Criticisms, comments, suggested changes, or topics for future coverage are encouraged, and should be forwarded to the Defense Intelligence Agency, Washington, DC 20301 (ATTN: DT).
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</tbody>
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Article I.
Key Developments in Soviet Metallurgy

William F. Marley
James R. Wamsley

SUMMARY AND ANALYSIS

Soviet metallurgy has achieved a high level of technical competence and is now recognized as a significant contributor to the world's technological base. Although the Soviets have excelled in capitalizing on foreign technical innovations, they have made significant indigenous advancements which have received broad acceptance throughout the world.

The items discussed in this article by no means cover all the Soviet achievements in metallurgy, but represent areas of significant advancement. When Soviet metallurgy technology is analyzed, many of the important developments appear to be related to metals processing. Although new or improved alloys are being developed, processing achievements are apparently being emphasized.

Notable advances in alloy development include the introduction of at least two Al-Li alloys for aircraft application and heat-resistant titanium alloys, including intermetallics with impressive elevated temperature properties. Achievements in steel technology include microalloying studies to affect increased mechanical properties and development of a high-strength nitrogen stainless steel to replace the more costly austenitic stainless steel.

Electroslag remelting and plasma arc remelting are widely used in the metals industry, and the Soviets have achieved prominence in both areas. Developments evolved from the basic electroslag technology include basic casting, hollow billet production, shape casting, and electroslag welding.

Plasma arc remelting is probably the most versatile of the refining processes. Soviet advances in this area have provided them with a production capability for the high-strength nitrogen stainless steels.

Thermomechanical treatments are recognized as a principal means to improve mechanical properties. These treatments are finding wide application in the metals industry.
Casting in an electromagnetic field for the production of aluminum and steel ingots is another Soviet achievement in process metallurgy. Commercial-size ingots with excellent surface characteristics are currently being produced.

The Soviets have certainly gained from exploitation of foreign developments, but have attained a respectable level of competence by their own indigenous accomplishments. At one time perhaps we could learn little from Soviet technology; however, because of their advancing capabilities, it is essential that we now maintain an increased awareness of their metallurgical developments.

DISCUSSION

Background

Soviet achievements in metallurgy cover a broad spectrum from the strong R&D programs for basic alloy development and materials analysis to materials processing for improving the production capabilities for high-quality materials. These programs are supported by the upper echelon of the government, which is technology oriented. Brezhnev, himself, was trained as a metallurgist. At least one-half of the Politburo and approximately three-fourths of the Council of Ministers have technology or industrial management backgrounds. The current Soviet hierarchy is cognizant of material needs and realizes the importance of maintaining a strong R&D capability. As a consequence, the Soviet metallurgical industry has achieved notable successes in alloy development and basic materials processing.

Alloy Development

While many Soviet alloys are identical to alloys developed in the West, the Soviets have made original contributions. Soviet emphasis on the Al-Li system has led to at least two new alloys. Alloy 01420, and Al-Mg-Li alloy is the lightest of all known aluminum alloys. Its application can reportedly provide a 10%-15% component weight reduction. The Al-Zn-Mg alloy system (US 7000 series) has focused on alloys strengthened by natural aging. Two alloys, 1915 and 1911, are now widely used; both alloys are medium strength (ultimate tensile strength ~55 to 60 klb/in²) and possess very good stress corrosion resistance properties.

New high-temperature titanium alloys have been introduced for gas turbine engine applications. Soviet research has led to the development of a family of high-strength, heat-resistant alloys known as the “ST” series. One of these alloys, ST-4, is in
production. If the reported claim for application of ST-4 up to 700°C for long service life is true, the alloy would be the most heat-resistant alloy yet developed.

The Soviets have embarked on a major research program to develop titanium base intermetallic compounds, even though brittleness characteristics are well known. Extensive research in the Ti₃Al intermetallic has culminated in development of at least one alloy, designated ST-5. The type of research in progress and the elevated temperature data being tabulated imply its potential application for gas turbine engine components.

Recent Soviet steel technology has focused on microalloying as a viable technique to effect increased mechanical properties. Microalloying with titanium, vanadium, tungsten, and/or niobium can be extremely useful in obtaining a ductile fracture microstructure, reducing the ductile-brittle transition, and providing steels with improved properties. In addition, active programs are underway to evaluate the effects of rare earth metal (REM) additions on steel properties. REM are known to affect inclusion morphology and lead to substantial grain refining.

A new nitrogen stainless steel (~1% N) has been introduced which could provide the Soviets with an excellent series of high-strength stainless steels at reduced cost since the nitrogen addition replaces nickel as a primary alloying element.

Materials Processing

Soviet metallurgical programs have provided major successes in metals processing and casting. Improving metal quality is a major Soviet goal, as stated in the current 5-year plan (1971-1975). The plan directs that "there will be a major effort to improve metal quality through the use of new production techniques." It stresses the importance of metals processing as the key to increasing production efficiency and, more importantly, achieving improved metal quality. Processing work on electroslag remelting has been vastly expanded, and plasma-arc remelting has been brought to production level.

Electroslag Remelting Technology (ESR)

The importance of ESR was recognized by the Soviets more than 25 years ago. With their strong technological base in electroslag welding, the Soviets were able to readily adapt the ESR process to production. Although the process is not an indigenous

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*The basic process was developed in the United States by R. K. Hopkins and patented in 1940, yet it was never widely accepted. Large capital investments in vacuum arc remelting equipment were a principal reason for an apparent (at the time) US lack of interest in ESR.
development, the Soviets have been very successful in scaling up the process to production and adapting it for 60-ton and larger steel ingots. Its wide utility and relatively low production costs led to rapid expansion of the ESR process throughout the sixties. Between 1958, when the first ESR steel ingot was produced, and 1969, Soviet annual production expanded to approximately 400,000 metric tons. The annual production rate is expected to reach 1 million metric tons by 1976.

The major advantage of the ESR process is the high purity levels that can be achieved. This high purity enables the materials to be heat treated to high-strength levels while retaining sufficient ductility and toughness to permit plastic deformation without excessive cracking or fracture. The increased fatigue strength and impact resistance makes ESR steels candidates for gun tube and armor plate application.

Some recent ESR technological developments introduced by the Soviets include: unique furnace designs, adaption of electroslag technology to casting ingots with improved metal-slag interaction, a capability to manufacture a multitude of shaped castings (production remelting of various types of hollow billets), electroslag welding, and thermomechanical treatment of ESR processed steels.

Furnaces

Current Soviet ESR production furnaces have evolved from a basic design of the mid-fifties. The original ESR furnace (Model R909, 0.5-ton capacity) was designed in 1956 and placed in operation in 1958. Since this original design, several furnace designs have evolved to meet the strong demands for larger ingots and requirements for higher quality materials. Shown in figure 1-1 is a general view of the Soviet ESR bifilar furnaces.

The USSR has expanded this basic technology to production capabilities for casting up to 150-ton ingots of variable shapes. The current state-of-the-art in large-capacity furnaces is exemplified in the three representative 150-ton systems listed in table 1-1.
Figure 1-1. General view of ESR bifilar furnaces of the U436 type.
### Table 1-I. Characteristics of ESR 150-Ton Furnaces

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Single phase</th>
<th>3 phase</th>
<th>EShP 150 7 electrode</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ingot size (mm):</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Height</td>
<td>3,900</td>
<td>3,900</td>
<td>3,900</td>
</tr>
<tr>
<td>Diameter</td>
<td>2,500</td>
<td>2,500</td>
<td>2,500</td>
</tr>
<tr>
<td>Movement of furnace parts:</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mold</td>
<td>Stationary</td>
<td>Stationary</td>
<td>Moving</td>
</tr>
<tr>
<td>Electrode</td>
<td>Moving</td>
<td>Stationary</td>
<td>Fixed</td>
</tr>
<tr>
<td>Ingot</td>
<td>Stationary</td>
<td></td>
<td>Stationary</td>
</tr>
<tr>
<td>Hollow ingots</td>
<td>--</td>
<td></td>
<td>Possible</td>
</tr>
<tr>
<td>area of electrode</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ratio</td>
<td>0.7</td>
<td>0.25</td>
<td>1.0</td>
</tr>
<tr>
<td>area of ingot</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Electrode:</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Number</td>
<td>1</td>
<td>3</td>
<td>7</td>
</tr>
<tr>
<td>Diameter (mm)</td>
<td>2,100</td>
<td>720</td>
<td>940</td>
</tr>
<tr>
<td>Length (mm)</td>
<td>5,600</td>
<td>15,500</td>
<td>3,900</td>
</tr>
<tr>
<td>Height of furnace</td>
<td>13,000</td>
<td>23,600</td>
<td>1,200</td>
</tr>
<tr>
<td>Weight of moveable part ton</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>of furnace during remelting</td>
<td>200</td>
<td>200</td>
<td>30-40</td>
</tr>
<tr>
<td>Electricity:</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Phases</td>
<td>1</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>Transformers</td>
<td>1</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>Capacity each transformer (kVA)</td>
<td>18,000</td>
<td>12,000</td>
<td>14,000</td>
</tr>
<tr>
<td>Power factor</td>
<td>0.3-0.5</td>
<td>0.75</td>
<td>0.90</td>
</tr>
<tr>
<td>Power consumption (kWh/ton)</td>
<td>1,700</td>
<td>1,400</td>
<td>900</td>
</tr>
<tr>
<td>Slag consumption (kg/ingot)</td>
<td>18,100</td>
<td>24,000</td>
<td>18,100</td>
</tr>
</tbody>
</table>

The single-phase furnace is a larger version of the original single-electrode design. Electric current from a welding transformer heats the slag bath by resistance heating, melting the consumable electrode. The molten metal is refined in the hot slag and then solidified at a controlled rate in the metal crystallizer. In the three-phase furnace, the
electric current travels between the electrodes, through the slag bath, to melt the consumable electrode. The additional heating provides an economic advantage by melting more metal at a lower cost. The seven-electrode model is a further refinement of the basic furnace design: it incorporates a moving mold and a stationary electrode, thus reducing the weight that must be moved during the remelting process. Both the electrodes and ingot are held by fixed supports during remelting. This technique allows for the production of hollow ingots with few modifications in the furnace structure.

The availability of these furnace types gives the Soviet designers the choice of the most efficient and cost effective method for production of specialty steels.

**Electroslag Casting**

The use of hot metal techniques, rather than cast electrodes, for remelting is another example of Soviet advancements in metal processing. The first developmental work in this area was conducted in the ESR facility of the Chelyabinsk Metallurgical Plant. The process involves a cold flux start, using a consumable electrode similar to the ESR practice, followed by the addition of liquid metal of the same composition as the consumable electrode until the crystallizer is filled. The electrode is withdrawn at the melt rate to allow it to maintain its relative position to the slag. The electrode is used to heat the slag and make up for shrinkage of the solidifying ingot. The resulting ingot has a metal purity approaching that of ESR steel, but has a macrostructure similar to an ingot cast in an iron mold. After a number of trials using several techniques, the use of preheated slag, liquid metal, and graphite electrodes evolved. The apparatus used is depicted in figure 1-2.

Note the short water-cooled crystallizer, conventional cast iron molds, the hot top lines with chamotte below the liquid level, graphite brick liner for the slag area, and graphite electrode for heating the slag. In addition, a corundum slab is floated at the metal-slag interface to break up the pouring stream. The pouring procedure involves:

- Putting melted slag in the crystallizer.
- Pouring liquid metal to fill the crystallizer.
- Floating the corundum slab at the slag-metal interface.
- Pouring metal to fill the mold.
- Maintaining slag temperature in the hot top until the ingot is 30% solidified.
- Complete cooling.
View A of figure 1-2 shows metal being poured into the mold after the corundum slab was added. View B shows the mold with the molten slag in the hot top. The molten slag is one part of the controlled solidification of the metal ingot.

This casting technique produces ingots containing one-fifth the sulfides and one-half the oxides of conventional pouring practice, and provides some reduction in anisotropy.

**ESR Hollow Billets Production**

The production of hollow steel billets is an inherently difficult problem because of the large number of engineering variables involved in the process. Consideration must be given to the mechanism for casting of the hollow billets, cooling requirements for the process, shrinkage problems encountered during cooling, removal of the mandrel, and the actual handling of the cast ingot. Examples of ESR-produced hollow billets are shown in figure 1-3.

Four techniques have been reported by the Soviets to counter the engineering problems:

- A segmented removable mandrel.
- A fixed tapered-bottom mandrel.
- A mandrel mounted on the crystallizer.
- A moving bottom-mounted mandrel.
Figure 1-3. Hollow tube billets obtained by the ESR method.
The segmented mandrel requires a complicated assembly, which has a limited life because of the high temperatures associated with the remelting process. The method does not provide a smooth inner surface because of the segment joints, and the concentricity of the billet may not be adequate. Also, a low electrode cross section or a specially cast electrode is required.

The use of a one-piece, hollow, tapered mandrel eliminates the costs associated with producing the segmented mandrel and provides an improved surface finish. This technique requires a special mold, however, with a hydraulic mechanism to remove the mandrel at the end of the heating cycle before the solidified steel has cooled and contracted.

The third method uses a movable mushroom-shaped head which is raised in the center of the crystallizer as the metal is added to the ingot. A solid electrode can be used, reducing electrode cost. The disadvantages are the need for a more complicated control mechanism to maintain the position of the mushroom head in relation to the ingot height and the concentricity problems which are inherent in a mandrel which moves during the remelting cycle.

In the fourth method, a center mandrel fastened to a moving crystallizer has advantages in the concentricity of the internal diameter and the cost of the mandrel, if the moving crystallizer technique has previously been adapted. The major disadvantages are the low ratio of electrode to ingot cross section ratio and the specially cast multiple electrodes that are required.

Electroslag Remelting for Castings

The electroslag process can be adapted for shaped castings. This permits production of high-quality parts with complicated shapes without an investment in forging equipment. ESR casting also reduces the amount of welding and allows the designers to eliminate longitudinal joints, thus providing a safer product.
The electroslag casting process either remelts a consumable electrode directly in the mold of a complex shape or in a special smelter. This is followed by gravity transfer of the liquid metal into the main mold. Overflowing of metal under the slag layer from the special smelter into the mold for the workpiece allows for casting of complex shapes. Previously formed parts can be used during ESR casting to form even more complex shapes. Figure 1-4 provides examples of shapes cast by this process.

Neg. 515856

Figure 1-4. Welded semicylinder made of cast electroslag metal.
Electroslag Welding

A major innovation in ESR technology is the application of the process to manufacture large hollow billets by electroslag welding. Examples of its use include welding of large gears, bearing races, reinforcing bands on large machines, billets for ring rolling, casting of separate quarter sections, and subsequent electroslag welding of these sections into rings. This technique is especially applicable to the manufacture of single billets and those with a height less than twice the diameter. It is possible to produce hollow billets of practically unlimited diameter by this technique. Figures 1-5 and 1-6 are examples of billets cast using this technique.

Neg. 515857

Figure 1-5. Hollow ESR billet for making bandages by expansion.
Plasma Arc Remelting (PAR)

Plasma arc remelting is receiving worldwide attention as a refining process because of its broad versatility. Where the other melting techniques require a specific environment, vacuum, slag, or inert gas shield, the PAR process can incorporate any of these characteristics in addition to high pressure. Equally important, the process permits a wide range of charge materials including scrap, sponge, and processed electrodes. Subsequently, a wide range of metals and alloys can be melted by the PAR process.
The above diagram (fig 1-7) is a schematic of a Soviet PAR system. The charge is a processed electrode which is slowly fed into a bank of plasma torches where melting occurs. Purification and solidification takes place in a mold attached to a withdrawal mechanism that lowers the ingot until the desired length is obtained. This and similar designs are used in production operations, the largest being a six-gun, 1.8-MW furnace.

PAR reportedly can be used to refine nickel, titanium, and high nitrogen steels. The versatility of the PAR method is particularly evident with the melting of their high-strength nitrogen steels; PAR is the first feasible method of producing such steels in large tonnage lots.
Thermomechanical Treatment

Thermomechanical treatment (TMT) is a viable processing technique for improving metal properties. This treatment consists of mechanically deforming a metal in various temperature regimes followed by selected cooling schedules.\textsuperscript{2, 3, 2, 4}

![Diagram of LTMT process](image1)

1. heating, aging accompanying tempering
2. low T deformation
3. high T deformation
4. aging (natural or artificial (one or two-step)
5. rapid cooling (quench)
6. second short term heating accompanying tempering

![Diagram of HTMT process](image2)

Figure 1-8. Typical sequences of the TMT process.
As illustrated in figure 1-8, two basic methods of TMT are employed, and combinations of both have been investigated. In high-temperature TMT (HTMT), the steel is deformed rapidly at a stable austenite temperature greater than the recrystallization temperature and followed by quenching. Low-temperature TMT (LTMT) involves deformation below the recrystallization temperature, again followed by quenching. Both treatments affect mechanical properties since they alter the microstructure. HTMT leads to higher ductilities and toughness, while LTMT provides for more significant increases in strength.

The effects of TMT are very dependent on steel composition, treatment temperature, and the extent of deformation. The Soviets have a large research effort to fully investigate optimum compositions of steels to be subjected to TMT. One result of Soviet HTMT research is the increase in tensile strength, impact strength, and particularly toughness of steels with 0.4% to 0.5% carbon, making these steels usable engineering material where heretofore their use was limited by their brittleness.

The Soviets were engaged in research on thermomechanical processing of electroslag remelted steels as early as 1968. From a synergistic standpoint, combination of both processes could allow wider application of ultrahigh-strength steels. A thermomechanical-treated steel that had previously been electroslag remelted would appear to be an excellent candidate for armor material, and equally important for applications where a high strength-to-weight ratio metal or alloy is needed, such as in aircraft structures.

Electromagnetic Field Casting

Another achievement in process metallurgy includes the development of casting in an electromagnetic field. Compared with the conventional continuous process, the new technique is claimed to be more economical because of higher metal yield, higher casting rate, and lower consumption of cooling water. The as-cast surface is so smooth and free of defects that machining is not required before further processing. The Kuybysshev Metallurgical Plant has recently introduced a technique of continuous casting of steel in an electromagnetic field with great success.

Presently, commercial-size ingots with surface characteristics superior to conventionally direct chilled cast ingots are produced. A schematic diagram of the casting apparatus is shown in figure 1-9. An inductor ring generates electromagnetic forces in the liquid metal as a result of eddy currents induced in the liquid metal. These currents and the axial component of the magnetic field of the inductor ring cause an electromagnetic
pressure on the surface of the liquid, thereby creating a situation where the liquid contracts and therefore does not come into contact with the mold.

As a viable process for casting aluminum alloys, the process won a 1973 State Prize. In 1970, the Soviets claimed that 70,000 metric tons of alloys were cast by electromagnetic casting and that by the end of 1971 close to 150,000 tons were to have been melted. More recent figures have not been reported, but, based on the above figures, this represents a significant level of growth.
Conclusion

The Soviet metallurgical industry has achieved notable successes in alloy development and metals processing in support of their technology base. Historically, they have depended on exploitation of foreign technology to support these requirements, but more recently, have emphasized indigenous programs for development of new or improved materials and for upgrading the quality of their material with advanced processing techniques. Examples of their most notable successes are ESR processing and related technology, PAR, new Al-Mg-Li alloy development, and electromagnetic field casting. These areas will continue to receive strong governmental support, with increased emphasis on industrial implementation of these technologies.

These technologies have had a major impact on development, design, and performance of Soviet systems; future developments will provide technologies to meet the superior performance requirements for use in advanced systems.
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Article II.

An Analysis of Soviet Efforts in the Fracture of Materials

Roy F. Frontani, PDTI,
Foreign Technology Division
Air Force Systems Command

SUMMARY AND ANALYSIS

An evaluation of Soviet activities in fracture mechanics indicates a broad-based, long-range effort is underway along two main paths: (1) problems of behavior of metals at low temperatures and (2) increasing the strength of materials. The effort appears to be more oriented toward engineering application than similar efforts in the United States. The Soviets have been actively studying the time dependence of fracture for over 10 years.

There is no indication that linear elastic fracture mechanics is employed in the USSR as a basic design methodology, although it is used in engineering sub-routines for estimating the reliability of materials. The notched-bar impact test is still the principal method of toughness evaluation for structural materials at low temperature. The instrumented impact test is rapidly gaining favor in the USSR, and continued use is anticipated. Emphasis on the more practical aspects of the fracture problem is concerned with reliability of structures and components, and of quality control. The effort on the fracture of polymers in the USSR has provided the basic groundwork for a comprehensive fracture program.

DISCUSSION

Introduction

The requirements for increased structural efficiency and, in turn, more efficient use of structural materials have become more demanding in recent years. As a result, the fracture of structural materials and reliable prediction of fracture are subjects of an intense and large R&D effort throughout the world. The goal of this effort is to incorporate meaningful fracture criteria into the design methodology for structural components. Recently, the problems associated with building structures in the northern regions have been addressed by the Soviets. Solutions to these problems are a prime consideration because of the large tonnages of materials involved.
Contemporary Soviet Philosophy

Soviet effort in the field of fracture of materials is being marshalled along two principal paths: (1) solving problems related to the behavior of materials at low temperatures and (2) increasing the strength of materials. The first effort is spurred by the accelerated Soviet program for construction of great industrial centers in the “North” to exploit their vast natural resources. Efforts along this line began seriously with the convening of the first All-Union Conference on the problems of “Increasing the Reliability and Service Life of Machines and Metal Structures Operating Under Conditions Associated with Siberia and the North.”¹,² This conference, held at Irkutsk in June 1968, has been followed by several others emphasizing other aspects of the problem. The second effort, directed at increasing usable strength of materials, is consistent with a world-wide concern to improve structural efficiency.

A review of Soviet publications indicates that basic research in the USSR is influenced to a greater extent by engineering problems than is evident in the United States. The program is closely related to engineering usage and, as a result, the definition of engineering problems is excellent. Fracture is conceived from more viewpoints than in the United States, with an apparent greater appreciation of the role of plasticity and localization of plastic flow. The role of the size effect in the fracture process is a subject of major interest and is receiving a significant amount of effort, both analytically and experimentally.

Time Dependence of Fracture

Considerable attention is being directed to fracture as a time-dependent phenomenon, conceived as the successive breakage of bonds through thermal activation. The leader in this field appears to be S. N. Zhurkov of the Ioffe Physicotechnical Institute in Leningrad. Zhurkov’s group points out that there are two entirely different approaches to the concept of strength: the static (mechanical), which conceives strength as an instantaneous loss of continuity which occurs at limit load, and the kinetic (thermal fluctuation), which involves a gradual accumulation of continuity defects (damage) with thermal fluctuations playing a decisive role.³ For some time, Zhurkov and other Soviet researchers have postulated the life of many types of materials, including metals, as being finite under static loading as a function of stress and temperature. This negates the viewpoint that, below $T_m/2^*$, creep deformation is negligible and therefore has no time dependence on fracture. This viewpoint was shaken by an investigation at the US Naval Research Laboratory that revealed a serious loss of load-bearing capacity for $\alpha\beta$ titanium alloys with elapsed time. Fracture toughness expressed as $K_{IC}$ values dropped 35% in a few days.

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¹ $T_m$ is the melting temperature.
Other Soviet activity on the basic aspects of the role of time dependency in the fracture process includes a phenomenological approach in the formation of the characteristics of stress-rupture strength, the development of a theoretical basis for life predictions, and a theory of long-time strength based upon the assumption that damage is characterized not only by its magnitude but also by its orientation in space.

Linear Elastic Fracture Mechanics

The discipline of linear elastic fracture mechanics (LEFM) is being investigated both theoretically and experimentally by Soviet researchers. Investigators have indicated that LEFM is the most precise method for comparing materials and that the resulting parameters can be used for design purposes. G. P. Cherepanov, a leading Soviet personality in the field of fracture, has reported extensively on Soviet research in LEFM. The Soviets possibly intend to exploit LEFM in real structural situations. There is little doubt that LEFM is being used in engineering considerations as sub-routines, particularly for estimating the reliability of a material in service in terms of factors such as its sensitivity to overloading, notching, natural cracks, and other stress concentrators.

Examples of the Soviet’s investigations of LEFM include: evaluations of ductility reserve for steels used in static and cyclic loading, methodology for determining $K_{IC}$ over a wide temperature range for structural steels, the effect of residual stresses, pressure vessel problems concerning the strength of pressure vessels in the presence of cracks, the evaluation of brittle strength, and the area of fractography. A typical example of the latter is the evaluation of aluminum and titanium alloys used in aerospace applications.

Notched-Bar Impact Test

The notched-bar impact test has been the traditional Soviet test for evaluating the engineering serviceability of metallic materials, and as a design requirement in terms of minimum values tolerated for a particular dynamic application. The standard specimen used in the USSR is the Mesnager "U"-notch coupon with a notch radius of 1 mm. Soviet interest and activity in the area of impact testing has continued at a high rate, particularly in support of the program to develop their northern regions. Currently, the Soviets are paying marked attention to the problems of resolving the total impact energy value into initiation and propagation energies, and the evaluation of cold brittleness in structural materials using the notched-bar impact test.
Livshits and Rakhmanov have reported on the role of initiation energy, \( (a_1) \) and propagation energy, \( (a_p) \).\(^{14}\) At least six methods are proposed (including LEFM) for partitioning the total energy absorbed in the test into initiation and propagation energy as a function of temperature. None of these methods is in universal use or agreement at this time. In an extensive analysis, Rauzin et al, discussed the methods of determining the impact toughness as a characteristic of the resistance to failure in the presence of notches and dynamic loads.\(^{15}\)

The instrumented impact test is presently considered the best overall method of evaluating the suitability of materials for structural applications at low temperatures, and it appears that it may be widely used in the expanded Soviet program of exploiting their arctic regions. On the other hand, LEFM is replacing the notched-bar test for the characterization of high-strength materials in plane strain situations at ambient temperatures.

**Fatigue**

Although the subject of fatigue is not within the precise scope of a review on fracture, the two areas are closely interwoven and are covered briefly in this section. The literature reviewed on fatigue is characterized by sophisticated theoretical work and a strong emphasis on fatigue in aircraft structures. One area receiving considerable emphasis is the relationship among energy criteria for crack propagation associated with melting and internal friction. Soviet fatigue predictions in aircraft structures are basically formulated by the Miner-Palmgren linear cumulative damage law and expressions for equivalent constant amplitude S-N curve for a particular spectrum loading regime.

**Engineering Application**

Emphasis on the more practical aspects of the fracture problems are concerned primarily with reliability of structures and structural components; consequently, statistical treatments of data are widely used to determine probability of fracture. The division of effort between steels and non-ferrous metals is considerable: in the ratio of 3 or 4:1. The evaluation of crack sensitivity in high-strength steel sheet is an area of high activity and interest. The optimization of the microstructure with respect to fracture resistance in low-alloy, high-strength steels was also the subject of a large program.\(^{16}\) Soviet interest in the thermal-mechanical treatment of steels for improvement of strength, ductility, and toughness has not abated and, more recently, has included efforts to improve the resistance to fracture as a primary goal.\(^{17}\)
In the area of non-ferrous metals, the focus is primarily on high-strength aluminum and titanium alloys for structural applications. Examples of these investigations were considerations of the initiation of longitudinal internal cracks in highly deformed structural aluminum alloys\(^1\) and the determination of fracture characteristics in \(\alpha\) and \(\beta\) titanium alloys, which showed that ductility is related to microstructure and phase composition and has no apparent relationship to the type of fracture surface.\(^2\)

Weldments, the major source of material defects in the fabrication of structures and components, are a continuing source of problems and, consequently, are the subject of numerous Soviet R&D programs. The quality control aspects of materials, structures, and structural components, particularly with regard to their processing (as in the United States) generates a large volume of engineering data.

**Fracture of High Polymers**

The fracture of polymers is particularly amenable to treatment by the kinetic theory of strength. Accordingly, the model of continuous disintegration of interatomic bonds through thermal activation is being applied to polymers by a group under the leadership of S. N. Zhurkov of the A. L. Ioffe Physicotechnical Institute in Leningrad. Zhurkov considers polymer fracture a four-stage sequence, one stage of which (formation of submicroscopic cracks by the grouping of "elementary" acts of rupture) is being studied intensively, by use of small-angle X-ray scattering.\(^3\)

Specific problems under investigation on the fracture of polymers include the study of the stress distribution at a notch by infrared spectroscopy,\(^4\) the effect of the state of stress in biaxial tension, and the amount of plasticizer on the strength envelope for polyvinyl chloride films,\(^5\) the kinetics of fracture of polystyrene under conditions of plane stress,\(^6\) and the scale effect in formed polystyrene.\(^7\) Limited evaluation indicates the Soviets have laid the foundation for basic portions of a fracture program for high polymers.

**Conclusion**

Soviet efforts on the development and application of LEFM are expected to increase during the next few years. The Soviets will use LEFM to replace the notched-bar test for the characterization of high-strength materials in plane strain situations at ambient temperatures. The notched-bar test will continue to be used to evaluate the suitability of materials for structural applications at low temperatures. Analysis of the low-temperature properties of materials will remain at a high level in support of the Soviet program to develop their arctic regions.
Time dependence of fracture has been of major concern and should continue to be studied in the development of a theoretical basis for fracture behavior and prediction. Continued development is also expected regarding fracture of polymers, particularly the high molecular polymers.
BIBLIOGRAPHY


Article III.
Soviet Coatings on Titanium Alloys
for Oxidation and Wear Resistance

R. C. Tucker, Jr., MAJ, USAR

SUMMARY AND ANALYSIS

The Soviets have developed a number of titanium alloys with sufficient strength at elevated temperatures to make their rate of oxidation a limiting factor in their use. They have, therefore, utilized molten metal immersion, pack cementation, electroplating, and plasma deposition to produce a variety of coatings to provide adequate oxidation resistance.

Single-stage silicide and aluminide coatings have been made by diffusion/reaction with the titanium alloys. More complex coatings of molybdenum silicide and nickel aluminide have been generated by first depositing molybdenum or nickel and subsequently reacting this layer with silicon or aluminum, respectively. In most of these systems, multilayer intermetallic coatings are formed.

In short term, isothermal oxidation of these coatings provided one to two orders of magnitude reduction in oxidation rate at $800^\circ$ to $1000^\circ$C, with the titanium silicides and aluminides being particularly outstanding. The coatings eliminated any internal oxidation of the substrates and the accelerated oxidation usually associated with the α/β transformation. Soviet efforts in the area of wear-resistant coatings reveal a continuing interest in nitriding. Both nitride layers and interstitial solid solution hardening have been studied, with an apparent preference for the latter. A Cr-Si coating applied by a pack cementation process has also been mentioned in the Soviet literature as improving wear resistance.

DISCUSSION

Introduction

The Soviets have devoted a great deal of effort to the production of titanium metal and the metallurgy required for its use. Their development of new alloys to meet specific requirements is based on phase equilibria and transformation kinetics studies. Apparently, sequentially developed binary, ternary, and then more complex alloys have been developed to determine the effects of each additive on the chemical and mechanical properties of the alloy.$^{1-4}$
Because of titanium's unusual corrosion resistance, the Soviet chemical process industry is deeply involved in the use of titanium, both for structural alloys and for coatings or cladding on other structural alloys.\textsuperscript{1,5} As is well known, titanium also has an unusually high strength-to-weight ratio and is therefore particularly important in the aerospace industry. In many applications, however, the titanium alloys must be coated to prevent excessive wear by fretting, galling, abrasion, or adhesion. For use at elevated temperatures, titanium alloys must be coated to prevent excessive oxidation.

Oxidation-Resistant Coatings for High-Temperature Titanium Alloys

Until recently, titanium alloys did not possess sufficient strength at elevated temperatures for degradation by oxidation to be of concern; e.g., titanium compressor blades and vanes in gas turbine engines were not used above 500° to 550°C. Soviet scientists, however, over the past decade, have developed a series of alloys with adequate strength at temperatures where oxidation becomes the life-limiting process of degradation, thus making the development of oxidation-resistant coatings imperative. These alloys include the predominantly $\alpha$ (hexagonal close pack) or $\alpha/\beta$ high-strength ST-1, ST-3, ST-4, ST-6, and IVT-1 alloys, the $\beta$ (body-centered cubic) corrosion-resistant AN-7 and TN-50 alloys, and the high-strength ST-5 alloy based on the Ti$_3$Al intermetallic.\textsuperscript{1,6} The AN-7 and TN-50 alloys are not only corrosion resistant, but retain high strength in the range of 800° to 1000°C. The ST alloys are of particular interest because of their potential use as compressor components in advanced gas turbine engines.

With the exception of ST-5, the ST series consist of highly alloyed $\alpha$ solid solutions of titanium with dispersions of Ti$_3$Al, Ti$_3$Sn, or TiCr$_2$. Their composition and phase transformations are shown in table 3-I (compiled from Ref. 6 and 7). The recommended service temperatures, table 3-II (Ref. 6), are consistent with the data of table 3-I. Table 3-III contains the instantaneous (ob) and longer term ultimate strength values of these alloys. It should be noted, however, that useful life in most gas turbine engines would be much greater than the longest period shown in table 3-III. A form of creep testing, centrifugal bending, used by the USSR is not directly comparable to US data; but, in comparison to other USSR alloys used in current compressor blading, the newer ST alloys, particularly ST-4, are far superior.\textsuperscript{6}

Oxidation of Uncoated Alloys

The oxidation of basic binary and ternary systems, as well as the more complex structural alloys, have been studied. A good example of the depth of these investigations is
the systematic study of the Ti-Al binary system and the Ti-Al-Sn-Zr systems that were apparently a part of the effort to understand the oxidation of the ST series of alloys. Such studies have included the determination of scale growth mechanisms as well as the diffusion of oxygen and alloying elements in the substrate during oxidation.  

Table 3-I. High-Strength, High-Temperature Titanium Alloys

<table>
<thead>
<tr>
<th>Designation</th>
<th>Composition</th>
<th>Phase* transformations, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>ST-1</td>
<td>Ti-Zr-Al-Sn</td>
<td>1040 990</td>
</tr>
<tr>
<td></td>
<td></td>
<td>β → α + β → α</td>
</tr>
<tr>
<td>ST-3</td>
<td>Ti-Zr-Al-Mo-Fe</td>
<td>1010 880 ≤700</td>
</tr>
<tr>
<td></td>
<td></td>
<td>β → α₂ + β → α₂ + α + β → α₂ + α + TiFe</td>
</tr>
<tr>
<td>ST-4</td>
<td>Ti-5Zr-9Al-5Sn-2Mo</td>
<td>1040 990 920</td>
</tr>
<tr>
<td></td>
<td></td>
<td>β → α₂ + β → α + α₂ + β → α + α₂</td>
</tr>
<tr>
<td>ST-5</td>
<td>Ti₂Al-?</td>
<td>1150 1000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>β → α₂ + β → α₂</td>
</tr>
<tr>
<td>IVT-1</td>
<td>Ti-7Mo-5.5Cr-3Fe-3Al</td>
<td></td>
</tr>
<tr>
<td>AN-5</td>
<td>Ti-Nb-Cr-Mo</td>
<td></td>
</tr>
<tr>
<td>AN-6, 6a</td>
<td>Ti-Nb-Cr-Al-B</td>
<td></td>
</tr>
</tbody>
</table>

*β = body-centered cubic solid solution of Ti.  
α = hexagonal solid solution of Ti.  
α₂ = Ti₃Al intermetallic ordered phase.

REFERENCES: 6, 7.

The AN-6 and -6a alloys are relatively stable β alloys which oxidize at a uniformly increasing rate with increasing temperature. The AN-5 alloy has lower aluminum and boron content than the AN-6 or -6a alloys and oxidizes at a higher rate at temperatures up to 850°C. At this temperature, the (TiNb)Cr₂ dispersion begins to dissolve, and the oxidation rate drastically decreases due to the increased homogeneity and decreasing oxygen diffusivity in the scale and alloy resulting from the effective addition of niobium.
Table 3-II. Maximum Recommended Service Temperature of the ST Series of Titanium Alloys

<table>
<thead>
<tr>
<th>Designation</th>
<th>Recommended Service Temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Short Term</td>
</tr>
<tr>
<td>ST-1</td>
<td>---</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>ST-3</td>
<td>750</td>
</tr>
<tr>
<td>ST-4</td>
<td>800</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>ST-5</td>
<td>800</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
</tbody>
</table>

References: 6, 7, 14*

Table 3-III. Ultimate Strength of the ST Series of Titanium Alloys*

<table>
<thead>
<tr>
<th>Test temperature</th>
<th>ST-1</th>
<th>ST-3</th>
<th>ST-4</th>
<th>ST-5</th>
</tr>
</thead>
<tbody>
<tr>
<td>600°C (1112°F)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Instantaneous</td>
<td>70</td>
<td>100</td>
<td></td>
<td></td>
</tr>
<tr>
<td>After 100 hours</td>
<td>25.5</td>
<td>16.5</td>
<td>33</td>
<td></td>
</tr>
<tr>
<td>After 1000 hours</td>
<td>14</td>
<td>11.5</td>
<td>21</td>
<td>37</td>
</tr>
<tr>
<td>After 2000 hours</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>750°C (1382°F)</td>
<td></td>
<td>75</td>
<td></td>
<td></td>
</tr>
<tr>
<td>800°C (1472°F)</td>
<td></td>
<td>50</td>
<td>75</td>
<td></td>
</tr>
<tr>
<td>900°C (1652°F)</td>
<td></td>
<td></td>
<td></td>
<td>40</td>
</tr>
</tbody>
</table>

*Defined as σb
The IVT-1 alloy oxidation rate is not affected by the lower temperature (450°C to 500°C) $\beta \rightarrow \alpha + \beta$ decomposition during aging, but it increases rapidly with the $\alpha + \beta \rightarrow \beta$ transformation at about 740°C. Moreover, at 900°C and above, a subscale of TiO forms between the TiO$_2$ major scale and the alloy. The subscale is a plane of mechanical weakness and would be expected to cause spalling during cyclic oxidation. The ST series of alloys apparently oxidizes in a similar manner, with a change in rate in the phase transformation temperature range.

Oxidation-Resistant Coatings

A number of techniques are used by the Soviets to produce oxidation-resistant coatings on titanium alloys—immersion in molten metal, pack cementation, and either electroplating or plasma deposition followed by a diffusion heat treatment. The Soviets used pack cementation to form titanium silicide coatings of Ti$_5$Si$_3$ with traces of TiSi$_2$ and molybdenum silicide coatings of MoSi$_2$ over Mo$_5$Si$_3$ on ST-4 alloys in one or two steps, respectively. This process consisted of packing the ST-4 specimens in pure silicon or molybdenum powder and heating them in a vacuum at 1200°C to 1300°C for 1 to 25 hours. The oxidation behavior of these coatings was compared with that of uncoated ST-4 as shown in figure 3-1.$^{14}$ A nickel aluminide coating of Ni$_3$Al and NiAl was also formed on ST-4 by the plasma deposition of first nickel and then aluminum, followed by a diffusion heat treatment of 1 hour at 800°C. Its oxidation performance was compared with that of the other coatings as shown in figure 3-2.$^8,^{14,15}$ The titanium silicide coatings formed a scale of TiO$_2$, the molybdenum silicide formed SiO$_2$, and the nickel aluminide formed $\gamma$-Al$_2$O$_3$ during oxidation.

![Graph showing oxidation in air at 1000°C](image)

**Figure 3-1.** Kinetic curves showing oxidation in air at 1000°C for ST-4 alloy before and after coating.
The titanium silicide coating provided the best protection of the three—better, in fact, than a nickel-20 wt% chromium alloy at 800°C and equal at 1000°C. It should be noted, however, that the test period, 25 hours, was extremely short relative to any useful service life: after a longer period of time, the nickel aluminide coating may provide superior protection because of the slower growth rate of Al₂O₃ compared to TiO₂ or SiO₂—particularly if α rather than σ Al₂O₃ is formed with longer periods of exposure. The use of any of the three types of coating eliminates any significant amount of oxygen diffusion into the substrate and the effects of the α → β phase transformation, which normally accelerates the oxidation of the uncoated alloy. The superiority of the titanium silicide coating was ascribed by the Soviets to the stability of the Ti₅Si₃ compound and the associated higher activation energy required for oxidation.

It is interesting that the oxidation rate for the MoSi₂ coating is much higher than that reported for solid MoSi₂ and that previous TiSi₂ coatings had less oxidation resistance than MoSi₂. (Ti₅Si₃ may convert to TiSi₂ at some point in the oxidation cycle due to loss of titanium to form TiO₂ depending on relative diffusion rates in TiO₂ and Ti₅Si₃, but no improvement in oxidation resistance was noted in the earlier work.) Moreover, TiO₂ is not normally considered a protective oxide scale because of the high diffusivity of oxygen through its lattice. The superiority of the titanium silicide coating over the molybdenum silicide, which forms an SiO₂ scale, may be due to the stability of the Ti₅Si₃, but it may also be improved by minor doping by the alloying elements in the ST-4 substrate. This possibility would, of course, be greater if the coating is formed by silicon diffusion inward rather than titanium outward. Only controlled experiments can determine conclusively the direction of diffusion. The high aluminium content of ST-4 may also reduce the oxidation rate of coated or uncoated specimens due to a discontinuous Al₂O₃ subscale formation.
Aluminide coatings on several alloys have been evaluated by the Soviets. Coatings produced by immersion in molten aluminum have been referenced in Soviet literature. In other work, TiAl$_3$ coatings were produced on the $\alpha/\beta$ alloy IVT-1 and $\beta$ alloys AN-5, AN-6, and AN-6a by first plasma-spraying the surface with aluminum and then effecting reaction/diffusion with the titanium by annealing at 800°C for 1 to 2 hours in vacuum. Oxidation tests of 50 hours duration were carried out at temperatures up to 1200°C for the AN-5, -6, and -6a alloys (as shown in Fig. 3-3) and 1000°C for the IVT-1 alloy (as shown in figure 3-4). In all cases, a $\sigma$ Al$_2$O$_3$ scale was formed. The significant increase in oxidation rate of IVT-1 associated with the $\alpha \rightarrow \beta$ phase transformation at 750°C to 800°C was effectively eliminated by the coating. The coated alloys apparently had better oxidation resistance than a Ni-20% Cr alloy. Again, the period of oxidation was very short relative to any useful service life. So long as the Al$_2$O$_3$ scale continued to form, however, the oxidation rate should be lower than that of Ni-Cr which forms a Cr$_2$O$_3$ scale.

![Graph showing oxidation rate vs. temperature](image)

Figure 3-3. Dependence of average titanium oxidation rates for titanium alloys and titanium-niobium based alloys on temperature.
The most common method of reporting oxidation in the Soviet references covered here was that of average oxidation rates for a fixed period of time. An attempt has been made in table 3-IV to compare the oxidation rates for several alloys and coatings at 800° and 1000°C. Unfortunately, some of the testing was done for 25 hours and the rest for 50 hours; for comparison purposes, therefore, it must be assumed the rate is constant. Considering the short period of testing, this may be misleading.

In only one instance was the initial period of scale formation treated separately. Oxidation during this period is normally very rapid, as is evident in table 3-IV, and can cause a significant error in the calculation of longer term, stable scale growth if it is not excluded. For comparison with the rest of the data, however, an average rate for 0 to 50 hours has been calculated in these instances. By comparing the 0 to 50 calculated average with the 5 to 25 measured average, this calculation illustrates the possible error that may result from extrapolation of the 0 to 25 or 0 to 50 averages commonly used if the initial period is not excluded.
**Table 3-IV. Oxidation of Coated and Uncoated Titanium Alloys**

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Coating type</th>
<th>Test period</th>
<th>Oxidation rate (mg/cm² h)</th>
<th>Reference No.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>800°C</td>
<td>1000°C</td>
</tr>
<tr>
<td>Pure Ti</td>
<td>None</td>
<td>0-25</td>
<td>0.052</td>
<td>5.29</td>
</tr>
<tr>
<td>Pure Ti</td>
<td>None</td>
<td>0-25</td>
<td>0.55</td>
<td>3.60</td>
</tr>
<tr>
<td>ST-4</td>
<td>None</td>
<td>0-25</td>
<td>0.21</td>
<td>5.06</td>
</tr>
<tr>
<td>ST-4</td>
<td>None</td>
<td>0-50</td>
<td>0.074</td>
<td>6.98</td>
</tr>
<tr>
<td>ST-4</td>
<td>None</td>
<td>0-25</td>
<td>0.043</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>0-5</td>
<td>-----</td>
<td>7.38</td>
</tr>
<tr>
<td></td>
<td></td>
<td>5-25</td>
<td>-----</td>
<td>3.02</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0-50</td>
<td>-----</td>
<td>3.46</td>
</tr>
<tr>
<td>ST-4</td>
<td>Si</td>
<td>0-5</td>
<td>0.014</td>
<td>0.296</td>
</tr>
<tr>
<td></td>
<td></td>
<td>5-25</td>
<td>0.0001</td>
<td>0.061</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0-50</td>
<td>0.0015</td>
<td>0.0845</td>
</tr>
<tr>
<td>ST-4</td>
<td>Si-Mo</td>
<td>0-5</td>
<td>0.05</td>
<td>0.688</td>
</tr>
<tr>
<td></td>
<td></td>
<td>5-25</td>
<td>0.004</td>
<td>0.085</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0-50</td>
<td>0.0086</td>
<td>0.145</td>
</tr>
<tr>
<td>ST-4</td>
<td>Ni-Al</td>
<td>0-25</td>
<td>-----</td>
<td>0.175</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0-5</td>
<td>-----</td>
<td>1.07</td>
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<td>5-25</td>
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<td>0.12</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0-50</td>
<td>-----</td>
<td>0.108</td>
</tr>
<tr>
<td>ST-5</td>
<td>None</td>
<td>0-50</td>
<td>0.012</td>
<td>2.185</td>
</tr>
<tr>
<td>IVT-1</td>
<td>None</td>
<td>0-50</td>
<td>0.160</td>
<td>2.51</td>
</tr>
<tr>
<td>IVT-1</td>
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<td>0-50</td>
<td>0.00</td>
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</tr>
<tr>
<td>AN-5</td>
<td>None</td>
<td>0-25</td>
<td>0.65</td>
<td>0.64*</td>
</tr>
<tr>
<td>AN-5</td>
<td>Al</td>
<td>0-25</td>
<td>0.00</td>
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<td>AN-6</td>
<td>None</td>
<td>0-25</td>
<td>0.047</td>
<td>0.247</td>
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<tr>
<td>AN-6a</td>
<td>None</td>
<td>0-25</td>
<td>0.047</td>
<td>0.224</td>
</tr>
<tr>
<td>Ni-20Cr</td>
<td>None</td>
<td>0-25</td>
<td>0.00</td>
<td>0.06</td>
</tr>
</tbody>
</table>

*Region of transformation (see text).*
Most of the data in table 3-IV has been extracted from small figures with concomitant errors in both plotting and extraction. Nonetheless, there appears to be a significant discrepancy in data for the uncoated ST-4 alloy. This possible discrepancy, however, does not alter the relative ranking of materials. It is apparent that at least an order of magnitude improvement in oxidation resistance is achieved with any of the coatings. If the effect of the difference in substrate alloys is ignored, the titanium aluminide coatings appear to be superior to the silicide coatings or the nickel aluminide coatings.

A relatively short period of oxidation testing is used in evaluating these coatings. It is much shorter than any useful service life and may be too short to establish that a single parabolic (stable solid-state diffusion) oxidation rate law is being followed. The mechanism of scale growth cannot be determined from available data.

A second point of interest is the lack of cyclic oxidation data: i.e., the effect of repeated, and particularly rapid, change from ambient temperature to the nominal temperature of testing. Cyclic oxidation can drastically change not only the rate of oxidation due to repeated exfoliation of the scale, but also the relative ranking of materials. Similarly, oxidation in high-velocity gas streams may be very different from that in relatively still air. Accelerated or catastrophic oxidation may occur as a result of surface contamination with various salts, but no mention of this type of testing was included in these references.

Another general area of concern is the lack of information on the effects of these coatings on the mechanical properties of the substrate. The effect on fatigue life may be significant since the intermetallics are relatively brittle, hence subject to cracking. Cracks formed, either as a result of foreign object damage or thermal fatigue, may propagate into the substrate, causing mechanical fatigue failure. In addition, cracks in the coating would lead to accelerated oxidation.

Comments on Oxidation Resistant Coatings

The Soviets have made impressive progress in the development of high-strength, high-temperature titanium alloys and coatings to protect them. Cyclic oxidation, hot corrosion, and mechanical properties may have been explored by the Soviets. The lack of apparent application of these coatings, however, may indicate continuing problems in one or more of these areas.
Wear-Resistant Coatings

Titanium has one of the greatest tendencies to gall of any of the structural metals. Alloying has little significant effect on this tendency, and it is usually necessary to provide some type of coating, shim barrier, or solid film lubricant in any situation involving metal-to-metal contact, particularly titanium-to-titanium. The Soviets have an active program in this area.

Coatings for Wear Resistance

A previous survey of titanium technology in the USSR refers to a Cr-50%Si mixture used to coat VT-5 alloys for wear resistance. Apparently, the wear resistance improved with increasing “chromosiliciding” time. If the process used to produce the coating was pack cementation and the ratio of chromium to silicon is the mixture of powder in the pack, the actual composition of the coating may not be Cr-50%Si, but rather another proportion of Cr-Si-Ti.

Nitriding titanium alloys for wear resistance has been described. Nitriding at 1 atm of pure nitrogen, as was done previously, results in a rather thick, two-zone nitride layer on the surface that inhibited diffusion of nitrogen into the substrate (in solid solution). In this work, a reduced partial pressure of nitrogen was used. Only a very thin nitride layer was formed and a much greater depth of nitrogen in solid solution achieved in a given period of time. The nitriding characteristics of the VT series of alloys and several experimental alloys were determined. Although the thicker nitride layers obtained earlier would have probably been very wear resistant, they were apparently considered too friable to be useful. Moreover, the part could not be machined to size after coating.

Both types of coating were applied to VT-5 alloy specimens and wear tested in an Amsler machine. There was no significant difference in the performance of the two, but the details of the results were not reported. It is not certain whether the thin nitride layer was penetrated and the nitrided titanium solid solution actually evaluated. The tensile and impact properties of the titanium core were unaffected, but the fatigue characteristics were not determined. A brittle coating or surface layer usually degrades the fatigue characteristics significantly. Continuing Soviet interest in nitriding titanium is evidenced by recent work on ion nitriding in a glow discharge. Only a 3- to 4-μm-thick nitride layer was formed, and nitrogen diffusion into the titanium occurs at two to three times the rate during simple furnace heating. No wear test results were noted, however.
Conclusions

This review of literature indicates a Soviet recognition of the oxidation and wear problems associated with titanium. Without coatings or alternative inserts or shims, the full utilization of the newer high-temperature, high-strength alloys cannot be realized.

These alloys will not be able to withstand the impact wear on mid-span stiffeners and platform edges or the galling and fretting in the root area without protection. Extensive effort by the Soviets in the wear behavior of titanium and its prevention should therefore be anticipated.
BIBLIOGRAPHY


Article IV.

A Study of Soviet Capabilities in
High-Performance Reinforcements and Composites

Franklin E. Wawner, Ph.D.

SUMMARY AND ANALYSIS

The Soviets are well aware of the advantages that "high-performance" composite materials* can provide in future military and commercial systems and are pursuing a research program that could provide them the materials for advanced structural requirements. With the realization that US scientists were involved in a fully committed effort in high modulus composite systems (which publically emerged in approximately 1969-1970 with the American Society for Testing and Materials (ASTM) and Society for the Advancement of Material and Process Engineering (SAMPE) conferences on composite materials), Soviet interest in this area increased as can be seen from the number of papers in the open literature. A strong theoretical effort was carried out in 1970 and 1971 on high modulus fibers and whiskers. The major emphasis in these works was on prediction of mechanical properties and thermal influence on residual stresses. Although appearing to be valid approaches the papers contain nothing revolutionary, and several are based on US predecessors.

Most of the characterization studies on boron and silicon carbide (SiC) continuous filaments are identical to those conducted in the United States during the period 1963-1966. One original contribution to basic studies on the fibers appears to be the internal friction studies on boron. The boron filaments are produced on a tungsten substrate 10 to 15 μm in diameter by chemical vapor deposition; the ultimate diameter of the filament is 90 to 100 μm. Production conditions for SiC filament are more explicit than for boron. Chamber length, production speed, deposition temperature, and reactant gas are all given.

*Within the context of this report, "high-performance" composites are defined as those which utilize reinforcements that possess one or more of the following properties: high strength (greater than 300,000 lb/in²), high Young's modulus (greater than 25x10⁶ lb/in²), low density, and good elevated temperature properties. Metallic and organic resin matrix materials, as well as continuous and discontinuous fibers and whisker reinforcements, are considered. Emphasis is placed on analysis of the "state-of-the-art" of composite systems that presently possess the most promising potential for usable materials components.
Average tensile strength data for boron and SiC filament produced and studied by the Soviets is approximately 200,000 and 100,000 lb/in², respectively, less than present US tensile strength data. Virtually all Soviet work in this area appears to follow earlier US work. This is emphasized by the suggestion to put a SiC layer on boron filament to serve as a diffusion barrier for metal matrix composites. In addition, the fact that the Soviets are investigating hybrid composite systems (such as combinations of glass-boron, etc.) and reinforcing commercial metal structures (such as aluminum I-beams with boron-aluminum tape) indicates that they are following US literature very closely and attempting to duplicate successful innovations. The Soviets are apparently having difficulties in producing sufficient boron filament of consistently high quality. The few papers containing composite data indicate their potential to produce structural bodies with properties similar to US materials.

Soviet capabilities in whisker-reinforced composites (with both polymer and metallic matrices) are probably equivalent to US capabilities. This can be attributed to continuing Soviet effort in whisker reinforcement technology. Mechanical property data presented in the Soviet open literature are generally equivalent to US data.

Standard problems encountered in international whisker composite technology—such as wettability, sorting, alignment and orientation, and bonding—appear to be major Soviet problem areas.

**DISCUSSION**

Conventional materials limitation for both critical military and commercial applications has brought an emergence in Soviet research programs for development of “high-performance” composite materials. Incorporation of these materials into structural components of new advanced weapon systems—i.e., aircraft, rockets, transport vehicles, etc.—could provide dramatic advancement in performance characteristics of military systems, with considerable weight savings in critical application areas.

As was the case in early Soviet work on glass-reinforced plastic (GRP), much emphasis has been placed on developing theoretical relations to predict material (system) behavior or to alter existing theories to determine if they are valid for high-modulus reinforcements or combinations of high- and low-modulus reinforcements (hybrid systems). Experimental evaluation of the composite systems appears to be in its infancy; however, considerable work has been accomplished on the individual reinforcements.
Boron and Silicon Carbide Filaments

Since the advent of continuous boron filaments in the United States in 1961, Soviet scientists have duplicated, with a 3- to 5-year gap, US technology for high-performance composite systems. Their programs have centered on making use of the high strength, high modulus, low density, and continuous nature of this material to utilize fabrication processes and techniques previously developed for other systems (specifically GRP).

Present strength and modulus values being obtained for selected high-performance composite systems and reinforcements are shown in table 4-1.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Strength (x10^3 lb/in^2)</th>
<th>Elastic Modulus (x10^6 lb/in^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Boron filament</td>
<td>395 (tensile)</td>
<td>56</td>
</tr>
<tr>
<td>SiC filament</td>
<td>240-300 (tensile)</td>
<td>65</td>
</tr>
<tr>
<td>B-A1 composite (33 vol% B)</td>
<td>430 (compression)</td>
<td>--</td>
</tr>
<tr>
<td>B-Al composite (VKA-1) (50 vol% B)</td>
<td>140-170 (tensile)</td>
<td>35.7</td>
</tr>
<tr>
<td>B-Epoxy composite (57 vol% B)</td>
<td>245 (flexure)</td>
<td>--</td>
</tr>
</tbody>
</table>

Boron has received considerably more emphasis in the USSR as well as in the United States than other continuous reinforcements, specifically SiC. Consequently, this section will primarily concentrate on boron.

Prior to 1970 there is very little in the Soviet literature on boron or SiC filaments. Most of the work concerning boron was accomplished by Samsonov who was primarily concerned with the chemical vapor deposition (CVD) of thin boron or boride layers onto metals to serve as protective barriers.

Gunyaev et al. published one of the first Soviet papers on boron filaments. These authors presented data on the strength, modulus, and strength distribution of boron filaments. It was not stated specifically whether they prepared their own filaments, only that the filaments were approximately 100 \( \mu \)m in diameter and were CVD onto a 10 to 12 \( \mu \)m tungsten substrate (typical US dimensions). Strength and modulus values quoted are
in line with those reported in the United States. A bimodal character was shown in the
strength histogram presented which indicates two active flaw sites. The authors attribute the
lower maxima to crystal defects on the surface of the filaments. In general, this work is a
duplication of information published in the United States in 1964. A stress-strain curve
presented in the paper is erroneous in that boron filaments are perfectly elastic up to
fracture. The curve shows a bend or change in slope, implying plastic deformation (this
probably results from the short gage length used).

The same authors investigated the influence of temperature on boron filaments in the
range of 20° to 600°C. Times of heat treatment were 1 to 1000 hours. Prolonged exposure
up to 200°C reportedly produced no effect on the strength of the filament. An interesting
effect was observed when filament was heated at 300° to 350°C for 1 hour. The tensile
strength was seen to increase by 10% to 12%; however, this was accompanied by a decrease
in flexural strength and modulus. Information to be presented later shows that there is an
internal friction peak in this temperature interval, which may be responsible for relaxation
of residual stresses in the filament and subsequent higher tensile strength.

Heat treatment above 400°C showed oxidation of the filament and finally the
formation of crystals on the surface, both of which led to decreased strength. Tensile values
were approximately 145,000 lb/in² after 1000 hours at 400°C. It was also noted that the
filament underwent a diameter decrease from 100 to 70 μm due to this treatment. This was
attributed to formation and evaporation of a continuous B₂O₃ film.

Prior to the initial studies on boron filaments, several Soviet scientists, and particularly
Samsonov, had conducted many studies on the borides. These works were primarily on
samples prepared via powder metallurgical techniques; however, CVD techniques were also
evaluated. Of particular significance is the work by Karev et al. who studied coatings of
boron on molybdenum formed by CVD.3 The primary thrust of this work was to compare
deposits from the H₂ reduction of BCl₃ and BBr₃. The authors synthesized their own BBr₃ by
reacting iodine vapor with boron at elevated temperature, a procedure which implies a desire
to find alternate routes for boron deposition.

Further techniques were studied for the production of boron by the reduction of
boron anhydride (B₂O₃) by magnesium in the presence of an oxidizer (potassium
chlorate).4 The basic reaction equation is

\[ B₂O₃ + 3Mg \rightarrow 2B + 3MgO \]
with the ultimate product being amorphous boron powder of 80% to 85% purity. The reaction was reportedly somewhat explosive, but produced better yields than earlier methods. The probable purpose of this work was to produce boron for further reaction with gaseous halides which would be used in subsequent CVD. It is possible that there is not an appreciable amount of basic starter gases (i.e., boron halides or hydrides) for boron deposition in the USSR based on the above works and on a statement that the Soviets are interested in purchasing boron filament from manufacturers in the United States.

Tarnopol'skii et al. prepared and evaluated composite systems of an epoxy resin matrix and either boron or carbon reinforcements. The composites were produced with fiber loading in unidirectional, orthogonal, and tridirectional orientations. The basis for this study was to analyze the anisotropic nature of high modulus reinforcement systems for comparison with GRP systems. Volume percent loading of reinforcement was 35% to 70%. Tests were made in tension, compression, and bending. In general, the boron composites gave strength data superior to the carbon composites (approximately three times greater for orthogonal composites in flexural tests), but modulus values were approximately the same. Unidirectional boron composites with 57 vol% loading gave average flexural strengths of 245,000 lb/in². More data are given for the boron-reinforced system than for the carbon system, implying more interest, but perhaps this is because of world interest at the time of writing. The authors recognized that distortion and misorientation of the stiff fibers were critical in composite preparation and influenced the mechanical properties considerably.

A review of the properties of modern composite materials and their application in air frame components was presented by Vol'mir et al. The paper is primarily a compilation of US and British data on high-performance reinforcements and organic and metal matrix systems. Nothing in the paper indicates Soviet developments in this area, but a strong interest is stated in theoretical aspects of understanding the systems. A further study by Tarnopol'skii mentioned the multitude of Soviet theoretical papers (100 in less than 2 years) on the mechanics of composite systems. He noted that valid numerical predictions were made on some property data; however, certain problems were encountered because of the noncontinuum nature of composites.

Because of poor interlaminar shear and transverse tensile strength in high-modulus fiber composites (i.e., boron and carbon), the author proposes three-dimensional reinforcement as a means of upgrading these properties. A weaving technique is presented to implement this approach but does not appear feasible, particularly for boron fibers of a rather large diameter (100 µm), which could not withstand the bending necessary for fabrication.
Other theoretical efforts have concentrated on predictions for factors such as cyclic loading, thermal cycling, fatigue, thermal stresses, and strength.\textsuperscript{18-23} Each of these studies was directed towards metal matrix composite systems with metal and/or high modulus inorganic reinforcements.

A study of a hybrid composite system was conducted by Skudra et al.\textsuperscript{24} The purpose was to determine the strength and stiffness characteristics of fiber glass-reinforced plastic that had additional reinforcement with high-modulus boron and carbon fibers and to determine methods of predicting these properties. This approach has been utilized in the US to attain higher modulus (stiffer) composites at a lower cost than using either boron or carbon alone. The Soviets obviously also realize the economic advantages to pursuing such “tailor-made” composite systems.

The majority of Soviet work on boron and SiC filaments, as reflected in the open literature, is concerned with metal matrices. An early paper by Tavadze et al. investigated the wettability of crystalline boron with molten metals.\textsuperscript{25} Copper, silver, gold, silicon, germanium, tin, and tin alloys were studied with regard to wetting (contact) angle on boron surfaces. This is probably initial work in determining compatible metal matrix systems for boron, particularly for preparation by casting and liquid infiltration from molten metals.

A series of characterization papers were published by Burykina, Dzyadykevich, et al. on the stability, compatibility, and reactions of boron and SiC with tungsten and titanium.\textsuperscript{26-29} This is an obvious thrust at analyzing these filaments as reinforcements for titanium matrix composites. The authors do not state their experimental procedure for producing boron filaments, only that the boron is deposited on 12-μm tungsten wire and that their ultimate diameter is 100 μm (typical US specifications). No mechanical property data are given for boron.

The paper does state how SiC filament was produced. Both single (12 inches long) and double (12 and 8 inches) deposition chambers were used, which were sealed by mercury electrical contacts. The reactant gas used was methyltrichlorosilane with a deposition temperature range of 1100° to 1220°C. Speeds of production were 3 to 5 in/min for the single chamber and 10 to 12 in/min for the double chamber. The resultant SiC fiber was 100 μm in diameter, had a density of 3.9 to 4.05 g/cm³, a tensile strength of 240,000 to 300,000 lb/in², and a modulus of $65 \times 10^6$ lb/in².

Data for the production of boron filament are not presented. This may imply that they are obtaining boron filament from other suppliers (boron filament is produced in the United
States, France, and Germany). The only statement concerning the deposition of boron is one in which the authors state the boron reacts vigorously with the incandescent tungsten substrate during the deposition process to form the intermediate phases $W_2B$, $WB$, $W_2B_3$, and $WB_4$. Production of boron filament in the US contains only $W_2B_3$ and $WB_4$, which implies the Soviets may be using a lower deposition temperature (nominal US deposition temperature is 1200° to 1250°C).

In one of the papers, the authors state that upon annealing at 900°C, the core compounds formed were $W_2B_3$ and $WB_2$ and that the fibers became embrittled and cracked. This implies that the fibers were produced by a low-temperature process, such as from the hydrides $B_2H_6$ or $B_3H_6$. Boron can be deposited from these compounds at approximately 500°C and form few reaction products between the tungsten substrate and deposited boron. With subsequent annealing, the boron will diffuse into the tungsten to form more boride phases, which will increase the volume of the core causing the boron sheath to crack.

The authors conclude that tungsten is not a satisfactory substrate material because of the diffusion and reaction to form borides. They propose either using another substrate material or developing a diffusion barrier to eliminate boron-tungsten interaction. What they do not realize is that boron undergoes an elongation of approximately 10% during deposition, which is closely matched by volume expansion in the tungsten due to the boride formation. If the borides did not form, then either the substrate would fracture causing a hot spot in the boron due to altered electrical conduction or the boron deposit would contain high residual stresses that would lead to cracking. The concept of using a diffusion barrier was considered in the United States in 1963 and quickly abandoned. After alternate substrates were evaluated, it was concluded that tungsten had the optimum properties.

Studying the interaction of SiC and tungsten during the production of SiC fibers, the authors observed very little reaction to form core compounds. On heating to 1200°C for a period of 12 hours, the Soviet authors report no further diffusion and reaction in the core.

In the studies of the reaction of boron and SiC with titanium, samples were prepared by the powder metallurgy process to produce composites that appear to have approximately 50 vol% boron filament (this was not stated). In general, the papers show that boron reacts with titanium at lower temperatures and shorter times than does SiC. Boron formed appreciable reaction products at 900°C, whereas SiC could reinforce titanium composites for prolonged operation at 1000°C and for short-time operation up to 1300°C. Reaction products in both systems followed a parabolic growth law or
\[ X^n = KT \]
Where \( X \) = layer thickness
\( K \) = reaction constant
\( T \) = time
\( n \) = integer going from 1 to 4, depending on the temperature

The authors of one paper\(^2\) suggested using a SiC coating on boron fibers to serve as a diffusion barrier for titanium matrix composites. (This has been in use in the United States since about 1968 under the trade name "Borsic").

Some fundamental studies on the mechanical properties of boron fibers were made by Zhigach et al.\(^3\) Tensile strength and fracture characterization were the main subjects of this study. Average tensile strength for a 1-inch gage length (accepted US specification) was 395,000 lb/in\(^2\) with a coefficient of variation of 39%. The strength distribution was unimodal, with the range of strengths varying from approximately 200,000 to 700,000 lb/in\(^2\). Included in the study was the influence of gage length on tensile strength.

The fracture surfaces obtained from the tests were placed in three categories: conchoidal, surface, and interfacial, with average strength levels given for each flaw. This study is essentially a repetition of US studies published in 1964-1966.\(^5\) The properties of the fibers used in this study are similar to those for US boron fibers produced in 1963. No mention is made of preparation technique for the filaments. The only parameter given is fiber diameter (90 \( \mu \)m) and length (500 m).\(^*\)

Some original basic studies on boron fibers by the Soviets include internal friction determinations.\(^4,5\) Relaxation maxima were found at -145°C and 380°C with activation energies of 0.64 eV and 2.36 eV, respectively. It is of interest to note that the peak at 380°C may have a connection with the increased strength (approximately 12%) obtained when filaments were heated to this temperature for 1 hour.\(^2\)

Mileiko et al. presented an interesting study on boron fiber-aluminum matrix composites.\(^6\) The samples were prepared by hot pressing in vacuum with unidirectional boron fibers separated by layers of D16 aluminum alloy (equivalent to US 2024). The composite specimens were made using boron fibers with three distinctly different average

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\(^*\)Present US boron filament is 100-\( \mu \)m diameter in continuous lengths (generally over 10,000 ft) with average tensile strength of 550,000 lb/in\(^2\) and coefficient of variation of 12%.
strength levels (257,000; 343,000; 470,000 lb/in²). The maximum compressive strength obtained in the composites was 430,000 lb/in² with 33 vol% boron fibers. Using higher fiber content did not increase the compressive strength.

The authors determined that the composite compressive strength was independent of the strength of the fiber used, showing that the major flaws in the filaments are primarily sensitive to tensile stress. They have also investigated the influence of fiber concentration on composite fracture characteristics in other studies.37

Other Soviet workers have fabricated a structural element for aircraft by pressure diffusion welding boron-aluminum tape of 50 vol% filament loading to an aluminum I-beam in the maximum stress bearing areas.38 The resulting structure gave flexural strength values of 140,000 to 170,000 lb/in² and 57,000 to 70,000 lb/in² at room temperature and after exposure to 40°C for 100 hours, respectively. Fatigue strength was also measured and seen to withstand 10⁷ cycles at stress levels of 57,000 to 70,000 lb/in². The elastic modulus at room temperature was 35x10⁶ lb/in². This B-Al composite system has been given the designation VKA-1. Calculations were made showing the superiority of using the composite material in that for certain components (jet longerons), weight could be reduced by 42% while increasing rigidity by 45% with no loss in strength.

This approach has been examined by US companies (Avco Corp.), and components based on strengthening of standard structural members by B-Al composites are being produced and used. The Soviet system above is probably a derivative of this work.

**Whisker-Reinforced Composites**

Analysis of "high-performance" composite systems must include whisker and single crystal reinforcements because of the strength values obtained for these systems. Whiskers represent the strongest form for any material. Tensile strengths experimentally obtained often approach values predicted on the basis of bond strength. When incorporated into a composite body, it is theoretically possible to obtain strength values approaching 1 million lb/in². Because of these high strengths, coupled with low density, high modulus, and high temperature properties of certain whisker materials, they offer tremendous potential for usage in aircraft, rockets, nuclear, and other military applications.

Prior to 1968, only a limited number of papers appeared in the Soviet literature on whisker reinforcements and composite systems. The predominant contributors to these
works were Bokshstein, Kishkin, and Svetlov, with emphasis on reviewing the general features of whisker reinforcements and composites, growth of the whiskers, and characterization of their mechanical properties.  

Aluminum oxide (Al₂O₃) whiskers received much of the early attention in these studies. Production of the whiskers was primarily through the high-temperature oxidation of aluminum or TiAl₃ powder in a hydrogen atmosphere. Tensile strengths obtained were as high as 2,300,000 lb/in² for samples tested in the optimum orientation. Great anisotropy in strength was noted to exist, depending on the particular growth orientation of the whiskers, tested. A strength dependence on diameter was noted for the whiskers, with strength decreasing as diameter increased.

Ductile metal whiskers such as copper, nickel, cobalt, and iron were also studied, but to a lesser extent than the higher melting and stiffer oxide, nitride, and carbide whiskers.

Bokshstein et al. conducted a series of elevated temperature strength tests on Al₂O₃ whiskers and found that whiskers which gave a tensile strength of 1,350,000 lb/in² at room temperature decreased to 865,000, 300,000, and 200,000 lb/in² at temperatures of 1100°, 1300°, and 1500°C, respectively. The authors noted that brittle fracture predominated up to 900°C, but transformed to plastic-type fracture above this range. A strength dependence on size was observed up to 1500°C.

Gunyaev and Sorina conducted a study on polymeric matrix composites using whiskers as reinforcements. Whiskers of Al₂O₃, TiO₂, AlN, SiC, and filamentary pyrocarbon (C) were synthesized for the study. Both unidirectional and randomly oriented reinforced composites were investigated. Impressive strength values were obtained both for the whiskers and the composites, as can be seen in table 4-II.

Studies to produce a refractory and chemically resistant composite material were conducted by Grosheva et al. Whiskers of mullite (3Al₂O₃•2SiO₂) were hot pressed into matrices of powdered AlN and ZrO₂. Mechanical property tests showed that resistance to thermal shock was much greater than for pure AlN, the compressive strength increased by a factor of four (12,000 to 50,000 lb/in²), and the impact strength more than doubled. The properties obtained make these composite systems particularly attractive as high-temperature materials.
Table 4-II. Mechanical Properties of Whisker Reinforcements and Composites

<table>
<thead>
<tr>
<th>Whisker Reinforcement</th>
<th>Matrix</th>
<th>Vol %</th>
<th>Strength (lb/in²)</th>
<th>Elastic Modulus x 10⁶</th>
<th>Reference</th>
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<tbody>
<tr>
<td>Al₂O₃</td>
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<td>2,300,000</td>
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<td>1,350,000</td>
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<td>TiO₂</td>
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<td>SiC</td>
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<td>1,140,000</td>
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<td>Filamentary</td>
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<td>Pyrocarbon (C)</td>
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<td>230,000</td>
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<td>Mullite</td>
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<tr>
<td>(3Al₂O₃·2SiO₂)</td>
<td>---</td>
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<td>243,000</td>
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</tr>
<tr>
<td>AlN</td>
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<td>1,000,000</td>
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<tr>
<td>Metallic and inorganic matrices</td>
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<td></td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>Silver</td>
<td>24</td>
<td>230,000</td>
<td>---</td>
<td>12</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>Aluminum</td>
<td>35</td>
<td>160,000</td>
<td>---</td>
<td>12</td>
</tr>
<tr>
<td>Mullite</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(3Al₂O₃·2SiO₂)</td>
<td>ZrO₂</td>
<td>15</td>
<td>36,000</td>
<td>---</td>
<td>16</td>
</tr>
<tr>
<td>Polymeric matrix</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SiC</td>
<td>Epoxy resin</td>
<td>14</td>
<td>115,000</td>
<td>---</td>
<td>12</td>
</tr>
<tr>
<td>SiC</td>
<td>Epoxy resin</td>
<td>25</td>
<td>110,000</td>
<td>---</td>
<td>14</td>
</tr>
<tr>
<td>C</td>
<td>Epoxy resin</td>
<td>50</td>
<td>74,000</td>
<td>---</td>
<td>14</td>
</tr>
<tr>
<td>TiO₂ *</td>
<td>Epoxy resin</td>
<td>11</td>
<td>30,000</td>
<td>4</td>
<td>14</td>
</tr>
<tr>
<td>AlN*</td>
<td>Epoxy resin</td>
<td>11</td>
<td>10,000</td>
<td>3.6</td>
<td>14</td>
</tr>
<tr>
<td>---</td>
<td>Epoxy resin</td>
<td></td>
<td>10,000</td>
<td>0.4</td>
<td>14</td>
</tr>
</tbody>
</table>

*Random orientation of reinforcing whiskers

Bokshteyn et al. point out the advantages as well as the many technological problems associated with producing composites from whisker crystals. In a 1971 paper, the authors state that the technology for producing Al₂O₃, SiC and Si₃N₄ whiskers is the most highly developed. Hot pressing in the presence of a fusible matrix phase and liquid (vacuum) impregnation are reported to be the best composite preparation techniques. An interesting statement is made concerning composite preparation by casting aluminum alloys containing small additions (5%) of Al₂O₃ whiskers; however, no details are given.
Whiskers of AlN have been grown by the reduction of Al₂O₃ by carbon in a nitrogen atmosphere.⁵⁶ The strength and modulus of the crystals was seen to be strongly dependent on geometrical orientation as well as on cross-sectional area (just as with Al₂O₃). Maximum strength and modulus values obtained were approximately 1 million and 50 million lb/in², respectively. The authors state that AlN whiskers would make good reinforcing elements for composite materials.

Although not specifically working with whiskers, a series of studies have been carried out at the Institute of Materials Science, Academy of Sciences of the Ukrainian SSR, on the reaction and wetting of refractory materials with various metals.⁵⁷⁻⁵⁹ Typical whisker materials such as Al₂O₃, AlN, BN, Si₃N₄, ZrB₂, and ZrN were investigated in contact with Fe, Co, Cu, Al, Ti, Zr, and Hf. The probable significance of these studies is to obtain a more thorough understanding of the compatibility of metal matrix-whisker reinforced composites for high-temperature applications.

Summary

Whisker reinforcements and composites have received considerable attention in the Soviet Union. Impressive property values have been reported for both the whiskers and the composites. Primary emphasis has been on oxide and ceramic type whiskers which are applicable for either metallic or organic matrices. Problems, such as wetting, sorting, alignment, and bonding, have inhibited large-scale development of components from these systems.
BIBLIOGRAPHY


4-16


Article V.
Soviet Glue-Welding Technology

Lowell Koht, 1LT, CmlC
J. Richard Hall, Ph.D.

SUMMARY AND ANALYSIS

Glue-welding is a Soviet innovation in joining technology which combines adhesive bonding and resistance welding. Two variations of the process have been reported: one is spot welding directly through a joint containing uncured adhesive; the other is the injection of liquid adhesive into a prewelded joint. Advantages of the process include reduced weight, enhanced joint corrosion resistance, increased static shear strength, and increased fatigue shear strength; it also offers economic advantages when compared to conventional joining techniques. Future applications range from ground mobility equipment to the production of structural components for high-performance aircraft and aerospace vehicles.

DISCUSSION

Introduction

A major Soviet innovation in joining technology is the development of a process known as “glue welding.”\(^1,2\) The process combines adhesive bonding and spot welding into a single process that takes advantage of the desirable characteristics of the individual techniques while offsetting shortcomings with complementary effects. Two variations of the process have been reported: spot welding directly through a joint containing uncured adhesive, and injecting a prewelded joint with liquid adhesive. The spot-welding and adhesive-bonding techniques, when employed separately, have vastly different joining characteristics and operational support requirements.

Spot welding has been used preferentially over riveting because of high productivity and ease of fabrication. To their disadvantage, spot welded structures are subject to corrosion between the overlapping elements. Spot welds also exhibit poor fatigue strength as

\(^\star\) Analogous operations in the United States are referred to as “Weldbond” processes.
a result of stress concentrations around the weld bead. Moreover, spot-welded structures cannot be anodized or subjected to other surface treatments because alkalies or electrochemical action may occur in the nonairtight joints.

Adhesive bonding provides joints that are airtight and exhibit good fatigue strength. As a result of the greater load bearing area, adhesive bonded joints show high lap shear strengths. Adhesive bonding, however, has the disadvantage of requiring expensive jigging and an inordinate amount of labor in laying up the bonded structures. Although adhesive joints exhibit high structural strength, they exhibit poor resistance to cleavage or nonuniform stress.

In the glue-welding method, stress concentrations around weld points and overlaps are reduced and redistributed, resulting in increased rigidity under stress and thereby increasing a structure’s load bearing capacity, particularly under cyclic load. The spot-weld operation is readily automated and the weld serves in place of expensive jigs, culminating in economical production.\textsuperscript{1,3} The corrosion-fatigue strength of glue-welded joints is improved over normal spot welds. For example, the application of an epoxy adhesive (Soviet designation VK-9) between joined steel plates improved the corrosion-fatigue characteristics so that the fatigue curve approached values of absorption fatigue.\textsuperscript{4} Figure 5-1 shows fatigue shear strengths of overlapped joints produced by several methods.

![Chart showing fatigue shear strengths](image)

**Neg. 516209**

Figure 5-1. Fatigue shear strength for the overlapping joint of duralumin.
Manufacturing Techniques

Production techniques and the quality of glue-welded joints are determined essentially by the adhesive compositions employed. Either or both of the following Soviet manufacturing techniques may be used, depending upon the properties of the adhesive and the geometry and engineering requirements of the desired article.

METHOD I – Spot welding through B-staged (uncured) adhesive can be subdivided into six basic operations: (1) preparation of the adhesive, (2) preparation of the faying surface, (3) application of the adhesive, (4) assembly of the parts, (5) spot welding, and (6) curing of the adhesive.

Welding through a layer of adhesive does not determine the width of the overlap or other design criteria, however, it does place some stringent requirements on the adhesive. Most liquid adhesives (excluding elastomer adhesives) are amenable to glue welding by this method. The major criterion is that the adhesive must be squeezed out of the contact area under electrode pressure in order to allow sufficient current flow for welding. The presence of unpressed adhesive leads to carbonization in the contact area. Gaseous evolution and void formation accompany carbonization, resulting in impaired bond strength and a permeable, glue-welded joint.

Although elastomer-based adhesives are readily pressed out of the contact site, they form dielectric films on the faying surfaces and inhibit the weld current flow which results in charring. To remedy these shortcomings, elastomer-based adhesive films have precut holes that correspond to the weld points.\textsuperscript{1,5} Although laborious, this technique is employed in special applications and is appropriate for both noncompressable and heat-resistant adhesives.

A further feature controlling Method I production techniques is the filler content, which affects the adhesive’s initial viscosity. Increased filler content is responsible for higher viscosities and increased difficulty in squeezing the adhesive from the contact point. A maximum viscosity of 0.12 second (cone viscometer) is recommended. Conversely, too low a viscosity allows adhesive flow from the joint, particularly if the gluing attitude is not horizontal.

In general, a welding machine with variable electrode pressure is recommended. Initially, pressure is applied to force the adhesive from the weld site, and low current is applied to warm films or high-viscosity adhesives. Welding current is then applied, followed by increased pressure to forge the hot nugget and prevent cracking.
Adhesives for this method must have low viscosity, long pot life, and form a dense, nonporous glue line upon curing. Solvent-containing adhesives were found to have the best penetrating and filling properties, but suffer from considerable shrinkage and reduced water resistance. Nonetheless, solvent-containing adhesives, such as Soviet FLAS (an epoxy-furyl-phenol-formaldehyde), appear to have found application. A lengthy curing schedule, with slow initial heat-up, is required to ensure a void-free glue line that may be safely anodized. Alloys subject to intercrystalline corrosion (naturally aged aluminum alloy D16T*) cannot be heated at high temperatures for long periods and therefore require twice the cure schedule (see table 5-I).

Table 5-I. Curing Cycle for FLAS Adhesive in Glue-Welded Joints

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Initial heat-up</th>
<th>Intermediate heat-up</th>
<th>Final heat-up</th>
<th>Total cure Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Naturally aged D16T</td>
<td>To 80°C in 2k; hold 1 h</td>
<td>To 120°C in 30 min; hold 30 min.</td>
<td>To 140°C in 30 min; hold 30 min.</td>
<td>5 hr (not including cool down)</td>
</tr>
<tr>
<td>Artificially aged D16T or alloy not subject to intercrystalline corrosion</td>
<td>To 80°C in 30 min; hold 30 min.</td>
<td>--------</td>
<td>To 160°C in 1 h; hold for 1 h</td>
<td>3 h (not including cool down)</td>
</tr>
</tbody>
</table>

For more fluid adhesives, the only pressure needed is that required to close the gap between the mating surfaces. Welding through adhesive requires a 10% to 20% decrease in welding current and an increase in the electrode pressure of 15% to 25%, depending on the adhesive used. The quality of the weld depends on the pulse duration. For short pulse durations with a rapid weld current build-up, the contact is not stabilized and the adhesive is not completely removed from the weld site. The Soviets suggest that welding through adhesive be performed where ambient temperature is greater than 15°C and relative humidity lower than 75%.

*US Analog is aluminum alloy 2024.
Inadequacies of the process (Method I) include incomplete removal of adhesive due to variations in the process, increased viscosity of the adhesive near the end of its working life, and completion of the welding operation during the working life of the adhesive. Furthermore, surplus adhesive frequently contaminates the electrodes, necessitating frequent cleaning or resurfacing of the electrodes. Welding through adhesives has drawbacks as a production technique, leading the Soviets to prefer the other variant of the glue-welding process (Method II) whenever feasible.

METHOD II — The second variation, introduction of liquid adhesive into the prewelded joint by capillary action, can be subdivided into six basic operations: (1) preparation of the surfaces for welding and bonding, (2) assembly of the parts for welding, (3) welding of the parts, (4) preparation of the adhesive, (5) introduction of the adhesive, and (6) polymerization of the adhesive. The advantage of this process is that the welding and bonding processes can be divided and mechanized.

Adhesives for this method must have low viscosity, sufficiently long pot life, and form a dense, nonporous glue line upon curing. Solvent-containing adhesive compositions were observed to have the best penetrating and filling properties, but suffer the disadvantage of shrinkage and reduced water resistance. Nonetheless, solvent-containing adhesives (Soviet FL4S, and epoxy-furyl-phenol-formaldehyde) appear to have found considerable application. A lengthy cure schedule is required to remove the solvent and insure a void-free glue line which may be safely anodized. Alloys that are subject to intercrystalline corrosion (naturally aged D16T) cannot be subjected to high temperatures for long periods and therefore require greater cure times (table 5-1).

Surface Treatments

To produce a clean weldable surface of uniformly low resistance (20-120 μΩ), surface treatment of the metals is necessary. To obtain high adhesive bond strengths for aluminum alloys the normal procedure involves the following steps: solvent wipe; alkaline degrease; rinse with warm (50°-60°C) water; rinse with cold water; chemical etch* and passivation; and finally rinsing and drying (60°C maximum).

*Etching bath composition: 110-115 g/l orthophosphoric acid, 0.8-1.5 g/l potassium dichromate. Etching is accomplished at 40°C for 10 minutes or 50°C for 7 minutes.
V. N. Shavyrin, among others, recommends abrading the metal surface after the chemical etch to roughen the surface and obtain good adhesion. Surface abrasion is followed by degreasing with acetone or ethyl acetate. B. B. Rubanovich reports that etching aluminum alloy in aqueous/molar sodium hydroxide and neutralization with 30% nitric acid results in a 50% reduction in bond strength as compared to the bond strength obtained by mechanical surface treatment (these results are contrary to US and other western findings).

Soviet Adhesives

D. A. Kardashov lists VK-1, VK-1M, VK-1MS, KS-609, FL4S, VK-7, and KLN-1 as adhesives that are useful in glue welding. An example of the variation in penetration depth of KS-609 into welded lap joints as a function of substrate thickness and time is shown in table 5-II. Tables 5-III and 5-IV show property data for some of the useful adhesives. Shear strengths of glue-welded joints at room temperature are shown in Table 5-V.

Table 5-II. Depth of Penetration of Glue KS-609 into the Clearance of a Joint Depending Upon Thickness of Sheets Being Welded

<table>
<thead>
<tr>
<th>Combination of thickness (mm)</th>
<th>Duration of holding (h)</th>
<th>Depth of glue flow (mm) according to position of sample in the process of holding</th>
<th>Horizontal</th>
<th>At an angle of 30° to horizontal</th>
</tr>
</thead>
<tbody>
<tr>
<td>3+3</td>
<td>1</td>
<td>20–25</td>
<td>20–25</td>
<td>25–30</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>20–25</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2+2</td>
<td>1</td>
<td>18–20</td>
<td>20–25</td>
<td>25–30</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>20–25</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.2+1.2</td>
<td>1</td>
<td>3–5</td>
<td>15–20</td>
<td>20–25</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>15–20</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.8+0.8</td>
<td>1</td>
<td>Almost none</td>
<td>In separate spots</td>
<td>8-10, local nongluing</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>In separate spots</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

5-6
<table>
<thead>
<tr>
<th>Designation</th>
<th>Type</th>
<th>Solvent</th>
<th>Pot life</th>
<th>Cure conditions</th>
<th>Heat temp</th>
<th>Glass transition temp</th>
<th>Heat resistance</th>
<th>Application</th>
<th>Suitability for high-temperature welding</th>
<th>Penetration depth</th>
<th>Resistance to flux ( \frac{N}{m} )</th>
<th>Viscosity ( \frac{N}{s} )</th>
<th>Resin type</th>
</tr>
</thead>
<tbody>
<tr>
<td>W-1</td>
<td>Epoxy resin with</td>
<td>none</td>
<td>8-10 h</td>
<td>100°C for 3 h or 150°C for 1 h</td>
<td>150°C</td>
<td>145 (DIN 53414, epoxy paper cleaned)</td>
<td>200-250</td>
<td>Yes</td>
<td>Yes</td>
<td>penetration up to 30 mm</td>
<td>Yes</td>
<td>No, high viscosity</td>
<td></td>
</tr>
<tr>
<td>W-1M</td>
<td>same as W-1 plus second hardener (polyethylene)</td>
<td>1 h</td>
<td>20°C for 18-20 h</td>
<td>20°C for 3 h or 150°C for 1 h</td>
<td>150°C</td>
<td>161 (DIN 53414, epoxy paper cleaned)</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
</tr>
<tr>
<td>W-10S</td>
<td>similar to W-1, contains at least 5% vinyl acetate polymer</td>
<td>1-1.5 h</td>
<td>100°C for 3 h or 150°C for 3 h</td>
<td>150°C</td>
<td>185 (DIN 53414, steel brush cleaned)</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td></td>
</tr>
<tr>
<td>W-7</td>
<td>Modified epoxy resin with basic hardener, used for bonding of stainless steel, etc.</td>
<td>1 h</td>
<td>25°C for 24 h</td>
<td>25°C</td>
<td>217 (DIN 53414, steel brush cleaned)</td>
<td>250°C</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
</tr>
<tr>
<td>W-9</td>
<td>Modified epoxy resin, from room temperature hardener, used for bonding of aluminum, steel, etc.</td>
<td>none</td>
<td>2.5 h</td>
<td>150°C</td>
<td>1.50 (DIN 53414, epoxy paper cleaned)</td>
<td>250-280</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
</tr>
</tbody>
</table>
### Table 5-IV. General Purpose Adhesives Suitable for Glue Welding

<table>
<thead>
<tr>
<th>Designation</th>
<th>Type</th>
<th>Solvent</th>
<th>Curing conditions</th>
<th>Bond strength (shear) kgf/cm²</th>
<th>Pot life</th>
<th>Max temp</th>
<th>Wt req’d for unit area g/m²</th>
<th>Suitability for Glue welding</th>
<th>Weld through method</th>
<th>Application after welding</th>
</tr>
</thead>
<tbody>
<tr>
<td>FLAS</td>
<td>Furfural-phenol formaldehyde polynvinyl formal, modified with ED-5 epoxy resin, dioctyl sebacate, aluminum filler</td>
<td>Alcohol-acetone (1:1) (65%-70% solids)</td>
<td>Hexamethylene diamine hardener (3 parts), 155°-160°C for 2 h</td>
<td>D16 at 20°C 116</td>
<td>8 h</td>
<td>60°C</td>
<td>200-300</td>
<td>Yes</td>
<td>Yes, 120-130 mm penetration</td>
<td></td>
</tr>
<tr>
<td>KS-609</td>
<td>Butyl methacrylate polymer in monomer solution; room temp peroxide cure</td>
<td>None</td>
<td>Room temp</td>
<td>D16F approx 70-80</td>
<td>1 h</td>
<td>60°-80°C</td>
<td>Applied as 0.5-1.0 mm layer</td>
<td>Yes</td>
<td>Yes, 20-25 mm penetration</td>
<td></td>
</tr>
<tr>
<td>KLN-1</td>
<td>Two part epoxy ED-5 epoxy resin (100 parts), active diluent DBT (20 parts), grade F polysulfide plasticizer (30 parts); polyethylene polyamine hardener</td>
<td>None</td>
<td>Room temp, or curing up to 80°C</td>
<td>D16 150-200</td>
<td>1.5-2 h</td>
<td>60°-80°C</td>
<td>150-200</td>
<td>Yes</td>
<td>Yes, 20-25 mm penetration</td>
<td></td>
</tr>
<tr>
<td>EPTs</td>
<td>Epoxy resin ED-5 or -6 with polyether acrylates M39 or T91, Portland cement filler, polyethylene polyamine hardener</td>
<td>None</td>
<td>Room temp; accelerated at 60°-80°C</td>
<td>EPTs: 80-150 (Duraluminium) EPTs: 50-80</td>
<td>1.5 h short term to 120°C</td>
<td>600-500</td>
<td>Yes</td>
<td>No, high viscosity</td>
<td></td>
<td></td>
</tr>
<tr>
<td>K153</td>
<td>Epoxy resin ED-5, polysulfide plasticizer, hexamethylene diamine, Portland cement</td>
<td>None</td>
<td>Room temp in 18 h; 80°C, 6 h; 100°C, 4 h. (annealed)</td>
<td>D16AT 180</td>
<td>1-1.5 h</td>
<td>60°-80°C</td>
<td>300-350</td>
<td>Yes</td>
<td>No, high viscosity</td>
<td></td>
</tr>
<tr>
<td>Glue</td>
<td>Method of fulfillment of glue-welded joint</td>
<td>Combination of thicknesses (mm)</td>
<td>Diameter of spot-weld nugget (mm)</td>
<td>Breaking load (kg)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>--------------</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Without glue</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FL4S</td>
<td>Application of adhesive after welding</td>
<td>0.6+0.6</td>
<td>3.5</td>
<td>470</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1+1</td>
<td>5</td>
<td>240</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.2+1.2</td>
<td>5.5</td>
<td>320</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.5+1.5</td>
<td>6.5</td>
<td>510</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>2+2</td>
<td>7.5</td>
<td>655</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>KLN-1</td>
<td>Application of adhesive after welding</td>
<td>1+1*</td>
<td>5</td>
<td>490</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.5+1.5*</td>
<td>6.5</td>
<td>560</td>
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<td></td>
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<td></td>
<td></td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>2+2</td>
<td>7.5</td>
<td>795</td>
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<td></td>
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<td></td>
</tr>
<tr>
<td></td>
<td>Welding through adhesive</td>
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<td>5.5</td>
<td>520</td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.5+1.5</td>
<td>6.5</td>
<td>575</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>2+2</td>
<td>7.5</td>
<td>770</td>
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</tr>
<tr>
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<td>Welding through adhesive</td>
<td>1.5+1.5</td>
<td>6.5</td>
<td>550</td>
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<td></td>
<td>2+2</td>
<td>7.6</td>
<td>780</td>
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<tr>
<td>VK-1</td>
<td>Application of adhesive after welding</td>
<td>0.6+0.6</td>
<td>3.5</td>
<td>615</td>
<td></td>
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<td></td>
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<td>1.2+1.2</td>
<td>5.5</td>
<td>725</td>
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<td></td>
<td></td>
<td>2+2</td>
<td>7.5</td>
<td>1025</td>
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<td></td>
<td>Welding through adhesive</td>
<td>2+2</td>
<td>7.5</td>
<td>1005</td>
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<td>VK-1MS</td>
<td>Application of adhesive after welding</td>
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<td></td>
<td></td>
<td>1.2+1.2</td>
<td>5.5</td>
<td>755</td>
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<td></td>
<td></td>
<td>2+2</td>
<td>7.5</td>
<td>1000</td>
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<td></td>
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<tr>
<td></td>
<td>Welding through adhesive</td>
<td>2+2</td>
<td>7.5</td>
<td>1045</td>
<td></td>
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<tr>
<td>VK-7</td>
<td>Application of adhesive after welding</td>
<td>1.5+1.5</td>
<td>6.5</td>
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<td></td>
<td></td>
<td>2+2</td>
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<td>7.5</td>
<td>745</td>
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Table 5-V. Shear Strength of Connections
of Duralumin D16T (overlap 25x25 mm)  (Continued)

<table>
<thead>
<tr>
<th>Glue</th>
<th>Method of fulfillment of glue-welded joint</th>
<th>Combination of thicknesses (mm)</th>
<th>Diameter of spot-weld nugget (mm)</th>
<th>Break Breaking load (kg)</th>
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</thead>
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<tr>
<td>VK-9</td>
<td>Welding through adhesive</td>
<td>1.5+1.5</td>
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<td>590</td>
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<td></td>
<td></td>
<td>2+2</td>
<td>7.6</td>
<td>820</td>
</tr>
<tr>
<td>K-153</td>
<td>Welding through adhesive</td>
<td>1.2+1.2</td>
<td>5.5</td>
<td>460</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.5+1.2</td>
<td>6.6</td>
<td>545</td>
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<tr>
<td></td>
<td></td>
<td>2+2</td>
<td>7.5</td>
<td>765</td>
</tr>
</tbody>
</table>

*Overlap 20x20 mm.

Economic Effectiveness of Glue-Welded Joints

In addition to high strength and operational characteristics, the production of glue-welded joints is claimed to be economically and technically advantageous. Spot welding with modern equipment is a rapid and relatively inexpensive process. The absence of a series of auxiliary operations and materials also reduces the prime cost of manufacture by the glue-welding process.

Modern structures used in transport engineering and construction must be airtight; therefore, it is expedient to compare a glue-welded joint with a hermetically sealed, riveted one. Soviet calculations show that the additional labor-input and prime cost of glue introduction during the manufacture of glue-welded joints are considerably less than analogous factors for hermetic sealing of riveted joints (see table 5-VI).

V. N. Shavryin states that when argon arc welding of large-dimensional, thin-sheet frame construction is used considerable warping occurs. Labor-consuming operations of correction are then needed. Consequently, the total technical economic effect from the use of glue-welded joints increases 2 times and more as compared to the data in table 5-VII.
Table 5-VI. Labor Input and Prime Cost of Manufacture of Constructions by Various Methods of Joining

<table>
<thead>
<tr>
<th>Type of joint</th>
<th>Prime cost of manufacturing 1 m seam (%)</th>
<th>Relative indices of manufacturing 1 m seam (%)</th>
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<tr>
<td>Spot welding</td>
<td>0.0693</td>
<td>Labor-input 100</td>
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<tr>
<td>Spot welding with use of glue</td>
<td>0.0815</td>
<td>Prime-cost 179</td>
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<tr>
<td>(glue-welded joint)</td>
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<td>Weight 110</td>
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<tr>
<td>Mechanized drilling and group pressing of riveting (nonhermetic)</td>
<td>0.0845</td>
<td></td>
</tr>
<tr>
<td>Mechanized drilling and group pressing of riveting with surface hermetic</td>
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<tr>
<td>sealing</td>
<td>0.1667</td>
<td></td>
</tr>
<tr>
<td>The same with intraseam hermetic sealing</td>
<td>0.2029</td>
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<tr>
<td>The same with two-zone hermetic sealing</td>
<td>0.2852</td>
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Thus, glue-welded joints are not only more durable, more reliable, and longer-lasting than other types of permanent joints, but are also claimed to be considerably more economical. This makes them very promising for application in various branches of machine construction.
Table 5-VII. Comparative Economic Factors for Welded and Glue-Welded Joints

<table>
<thead>
<tr>
<th>Combination of thickness (mm)</th>
<th>Variants of joints</th>
<th>Expenditure for 1 m seam (kopecks)*</th>
<th>Relative expenditure for glue-welded joint (%)</th>
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<tr>
<td></td>
<td>Argon arc welding</td>
<td>Argon arc welding</td>
<td>Glue welded</td>
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<tr>
<td>3+3</td>
<td>Single-pass butt</td>
<td>69.783</td>
<td>25.946</td>
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<tr>
<td></td>
<td>Single-row butt</td>
<td>102.234</td>
<td>25.946</td>
</tr>
<tr>
<td></td>
<td>Two-way</td>
<td>69.783</td>
<td>53.166</td>
</tr>
<tr>
<td></td>
<td>Single-pass butt</td>
<td>102.234</td>
<td>53.166</td>
</tr>
<tr>
<td></td>
<td>Two-way butt</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2+2</td>
<td>Single-pass butt</td>
<td>51.859</td>
<td>22.224</td>
</tr>
<tr>
<td></td>
<td>Single-row butt</td>
<td>75.699</td>
<td>22.224</td>
</tr>
<tr>
<td></td>
<td>Two-way butt</td>
<td>51.859</td>
<td>39.453</td>
</tr>
<tr>
<td></td>
<td>With sheet cover plate</td>
<td>75.699</td>
<td>39.453</td>
</tr>
</tbody>
</table>

*One one-hundredth of a ruble, equal to $0.0136 at current exchange rate.

Fatigue Strength

Rather extensive Soviet investigations have been conducted on the effect of an adhesive injected into a welded joint.\textsuperscript{1,4,10-12} Marked improvement was found when glue-welded joints were compared with spot-welded joints. The degree of improvement was dependent on the type of joint and geometry of the spot-welding pattern, apparently because of differences in the redistribution of stresses within the joint.
Thus for glue-welded, single-row joints, fatigue strength is 1.5 to 2 times higher than for welded joints (axial extension by asymmetric cyclic loading, factor 1.0×10^6 cycles). Values were 3 kgf/mm² for KLN-1, about 4.5 kgf/mm² for KS-609, and 4.5 kgf/mm² for VK-1 (fig 5-2 to 5-4).

For a two-row overlap joint, fatigue strength for glue-welded joints is 2-2.5 times higher than for welded, about 4 kgf/mm² for KLN-1, 6.7 kgf/mm² for VK-1MS, 4.5 kgf/mm² for FL4S, and about 5 kgf/mm² for VK-9. In a similar welded joint with VK-1 this value is 6.5 kgf/mm² for an adhesive-bonded joint (VK-1) 6 kgf/mm², and for a riveted joint, about 2.5 kgf/mm². Fatigue strengths using KS-609 are shown in figure 5-5.

Table 5-VII represents fatigue strength data obtained as a result of comparative tests of welded, glue-welded (glue KLN-1 introduced after welding), and riveted structural elements made from alloy D16T during asymmetric axial extension with the frequency of repeated loading of 200 c/min on the basis of 10^6 cycles. The working load on all joints was changed within limits of 750-2000 kg. It is clear that fatigue strength of glue-welded structural elements is more than 2 times higher than the strength of welded joints and 30% higher than riveted.

![Graph](image)

**Neg. 516212**

**Figure 5-2. Fatigue strength of single-row overlap joints of alloy D16T (1.5+1.5 mm) (VK and KLN adhesives).**

5-13
Figure 5-3. Fatigue strength of single-row overlap joints of alloy D16T (1.5+1.5 mm) (VK adhesive).

Figure 5-4. Fatigue strength of single-row overlap joints of alloy D16T (1.5+1.5 mm) (KS adhesive).
Figure 5-5. Fatigue strength of two-row overlap joints of alloy AMg6-1(2+2+2 mm) butts with bilateral flat cover plate.
Table 5-VIII. Number of Cycles Before Failure of Structural Elements Made From Alloy D16T (sheet $\delta=1$ mm+profile $\delta=2$ mm)

<table>
<thead>
<tr>
<th>Diameter of nucleus of point, rivet (mm)</th>
<th>Joint</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Welded</td>
</tr>
<tr>
<td>5.45</td>
<td>$47.6 \cdot 10^2$</td>
</tr>
<tr>
<td>4</td>
<td></td>
</tr>
</tbody>
</table>

CONCLUSIONS

The Soviet Union has refined the glue-welding process to a high degree and exhibits confidence in the resulting products. The process is used extensively in large Soviet transport aircraft and rotary-wing aircraft. Should the Soviets obtain greater sophistication in high-temperature, high-strength adhesives it is likely that the glue-welding process would be incorporated into supersonic aircraft and spacecraft production.


Article VI.
Material Composition and Construction of
Some Soviet Optical Apertures
Charles A. Petschke

SUMMARY AND ANALYSIS

Soviet materials engineers have selected several types of compositions for use in window construction. The primary materials used are organic glasses,\(^1\) silicate glasses, and combinations of the two.\(^2\)

Acrylic glasses have found extensive application in Soviet aircraft construction.\(^3\) These organic glasses are usually copolymers of acrylic and methacrylic acid derivatives, the most common being poly(methyl methacrylate) organic glass sheets. These rigid isotropic organics are designed for bearing tensile loads.

In addition to acrylic glass, windows of pressurized cabins are currently constructed of oriented organic glass and organic laminated safety glass. The microstructure of the oriented organic glasses originates from drawing organic glass in a highly elastic state (above the flow point). During this drawing, molecules are oriented with the direction of the action of external force, which leads to increased strengths and toughness.

Common organic laminated safety glass, which is used for optical apertures, consists of two organic glass sheets bonded to a transparent butvar film, and possesses properties nearly identical to ordinary (unoriented) organic glass. The advantage of safety glass is the highly local nature of massive failure upon impact and penetration. The common Soviet laminated organic glass (designated OT-16) is composed of two glass sheets bonded with butvar [poly(vinyl butyral)].

Inorganic glasses for transparent aperture materials are based on silicate and quartz glasses in the form of laminated safety glass. The most common form consists of two sheets of inorganic glasses bonded to a middle layer of butvar film. Silicate triplex is the common term for safety glass of this type. The glass plates protect the cementing layer from detrimental exposure; should the transparency be damaged, the layer binds glass fragments together, thereby improving the safety properties. Triplex may be used for load bearing (multiple plates) or non-load bearing (two sheets) purposes.

\(^{\text{1}}\)The Soviets use the term "organic glasses" to refer to transparent polymeric materials.
DISCUSSION

Organic Glass

Organic glasses are usually copolymers of acrylic and methacrylic acid derivatives, the most common being poly(methyl methacrylate) organic glass sheets. This material exhibits the usual desired properties of high transparency with respect to visible and ultraviolet rays, good moldability, high strength, good dielectric characteristics, and oil, gasoline, and water resistance. It is also easily machined and bonded and is produced in transparent/colorless, transparent/colorable, and opaque forms. Organic glass can be vitreous, highly elastic, or viscous, depending on the temperature of use. It softens on heating and hardens on cooling. These materials have found extensive application in Soviet aircraft construction.

Organic glasses with the Soviet designations SOL and ST-1 are produced commercially. SOL is a plasticized poly(methyl methacrylate). Aviation grade SOL organic glass is available in sheet form with a thickness range of 2-20 mm. SOL is molded at 105°-150°C, and is bonded at 140°-145°C for 20-30 min at pressures of 0.5-1.0 MN/m² (5-10 kgf/cm²). Bonding is accomplished with a dichloroethane-based adhesive. SOL is used for both pressurized and nonpressurized aircraft cabins, and is designed for operating temperatures between -60° and +140°C.

Aviation grade organic glass type ST-1 is composed of unplasticized poly(methyl methacrylate) with the addition of Phenyl salicylate. It is used in both pressurized and nonpressurized aircraft cabins, and is designed for the -60° to +140°C temperature range. Bonding to other materials is accomplished using dichloroethane-based adhesives.

Two other Soviet organic glasses are designated type 2-55 and type T2-55. Heat-resistant material 2-55 is a linear structure copolymer based on methyl methacrylate (MMA) with a photostabilizing component added. Type T2-55 is a thermostabilized copolymer whose distinguishing structural feature is the presence of infrequent macromolecular linkages, and which has thermostabilizing and photostabilizing components added. The partial cross-linking in T2-55 glass makes it more thermostable than type 2-55, although both possess similar basic physicochemical characteristics. The viscous region for 2-55 is about 200°C; T2-55, which can be utilized effectively to 240°C, has a viscous region that is greater than 300°C.
In the vitreous region, both 2-55 and T2-55 are rigid isotropic materials capable of bearing relatively high loads without material deformation (up to about 370 kg/cm² ultimate tensile strengths at 100°C). Although the strength of these glasses drops as the temperature rises, it remains sufficiently high up to the vitrification point of 130°C for long-term operation in this temperature region. The behavior of 2-55 material in the elastic state is similar to that of SOL and ST-1 in the glassy state. A distinctive feature of the thermostabilized glass T2-55 is its limited deformability in the region of 130°C. T2-55 exhibits a lower tensile elongation in the highly elastic state than non-thermostabilized organic glasses.

Oriented Organic Glass

In addition to organic glass, windows of pressurized cabins are currently constructed of oriented organic glass and organic laminated safety glass. Ordinary organic glass is quite brittle, especially under conditions of one-sided pressure gradient in cabins. The microstructure of oriented organic glasses originates from drawing organic glass in a highly elastic state (above the flow point). During this drawing, molecules are oriented with the direction of the action of external force, which leads to increased strengths and toughness.

Uniaxial drawing strengthens organic glass only in the direction of orientation, and the mechanical property values are lowered somewhat in the perpendicular direction, as compared to ordinary organic glass. Biaxial (plane or radial) drawing leads to hardening the material in any direction within the plane of the sheet.

Transparent components fabricated from biaxially oriented organic glass have increased static strength and local resistance to massive defeat during penetration. Optical properties of oriented organic glass are comparable to those of ordinary (unoriented) organic glass. DOR-16 is the designation for a common Soviet biaxially oriented organic glass used for cabin windows in high-altitude aircraft. It is composed of poly(methyl methacrylate), supplied in sheet form with thicknesses to 10 mm and widths to 190 cm, and designed for operating temperatures between -60°C and +60°C. The linear dimensions of oriented glass are stable at 15°C to 20°C below the softening temperature. After complete heating, oriented and unoriented organic glasses have the same maximum working temperatures: 60°C for SOL glass, 80°C for ST-1 glass, and 100°C for 2-55 glass.

Oriented glasses are molded by methods similar to those used for unoriented glasses. This consists of preliminary bending below the softening temperature, and then molding above the extension temperature, preventing material shrinkage by rigidly fastening the
organic glass blank around its circumference. The best method for producing windows based on organic glass is the "composite technique," which provides combined extension and molding of the material. In comparison with similar components of unoriented organic glass, windows made of oriented glass have a static strength of 1.5 to 2 times higher, superior shattering resistance under impact loads, and higher long-term static strengths (surviving more than 1000 hours at 175 kg/cm² stress and 80°C).

Laminated Safety Glass

Common organic laminated safety glass, which is used for optical apertures, consists of two organic glass sheets bonded to a transparent butvar film and possesses properties nearly identical to ordinary (unoriented) organic glass. The advantage of safety glass is the highly local nature of massive failure upon impact and penetration.

Optical deficiency of organic laminated safety glass is due to a tendency of this type of glass to form bubbles within the bonding layer when heated prior to molding. The probable cause of the bubbles is the presence of moisture in the butvar [poly(vinyl butyral)] film. During the manufacture of laminated organic glasses, moisture can be introduced into the bonding layer by insufficient drying of the film or by absorption of moisture from the air. The less moisture contained in the film, the fewer number of bubbles form in the molding process. Development of a hot-melt material compatible with the organic glasses, such as that developed by the US Army Materials and Mechanics Research Center for transparencies, has not been disclosed by the Soviets. This type of material could be used in glass-polycarbonate laminates in lieu of butvar adhesives, and would eliminate some optical problems.

Soviet laminated (triplex) organic glass OT-16 is composed of two sheets of organic glass each 4-6 mm thick, and bonded with a 2 mm thick butvar film. OT-16 is fabricated in sheet form with many different dimensions, is easily machined, can be welded, bonded, and molded, is designed for use in pressurized aircraft cabins, and has a temperature range of -60° to +60°C.

Inorganic Glasses

Transparent aircraft materials are based on silicate and quartz glasses in the form of laminated safety glass. The most common form probably consists of two sheets of inorganic glasses bonded to butvar film as a middle layer.
In those applications where canopy and port transparency must be maintained under high Mach number conditions, the Soviets employ high-strength, stress-relieved plate glass or glass laminated by polymeric interlayers bonded by adhesives. Special heat-resistant zinc-borosilicate glasses have been developed.

**Silicate Triplex**

Silicate triplex consists of glass plates cemented together. It is used for glazing automobiles, aircraft, helicopters, railroad rolling stock, test stands, etc. The glass plates protect the cementing layer from detrimental exposure; the layer binds glass fragments together if the transparency is damaged, and therefore improves the safety properties.

Silicate triplex not intended for load-bearing purposes is composed of two glass sheets of identical dimensions with an ultimate strength up to 300 kg/cm². The plates are cemented by a poly(vinyl butyral) film (butafoil) with a thickness of 0.5-1 mm. The total silicate triplex becomes 5-6 mm thick. Plates of identical thickness, which are not subjected to hardening (quenching), can be used for components of intricate configuration and large overall dimensions (curved automobile windshields, for example). This triplex is designed for use between -60°C and +60°C.

Silicate triplex intended for load-bearing purposes is somewhat different. One of the glass plates (usually the one facing the interior of an aircraft cabin) is thicker to carry maximum stresses. The greatest stresses that arise in both plates of the triplex should not exceed the overall allowable stresses however. The second glass plate is usually 4-5 mm thick. Load-bearing silicate triplexes are made from hardened glass sheets that have up to 800 kg/cm² ultimate tensile strength (UTS) for plates 4-5 mm thick, and up to 1200 kg/cm² UTS for 6-20 mm thick plates. The cementing layer is not less than 2-3 mm thick.

The use of transparent polymers with high thermal stability [nonplasticized poly(vinyl butyral) film, organosilicon compounds, etc.] extend the upper limit of the working temperatures. Silicate triplex subjected to highly variable service temperatures is composed of hardened heat-resistant glass with an expansion coefficient of $45 \times 10^{-7} / °C$ or smaller.

The basic bonding and installation of silicate triplex glass involves assembly or stacking of the components (cold and hot adhesive in vacuum, and liquid or gas pressing in autoclaves). Maximum evacuation of air from the triplex stack, relaxation of internal stresses in the cementing layer, and adhesion of materials onto the glass surfaces being bonded are all required for good triplex fabrication. Temperature and pressures must be closely controlled also.
CONCLUSION

This literature review indicates that Soviet engineering materials used for optical apertures are similar to those in the West. Both organic and inorganic (silicate) glasses are widely used for windows of airplanes, helicopters, automobiles, etc. Limited uses are found in building construction and illumination applications. Laminated glasses composed of two or more silicate glass sheets bonded together by a butvar film, plus one or more organic glass sheets bonded by a methyl methacrylate film, find application as bulletproof apertures. All Soviet aircraft and ground vehicles use one or more of these optical materials in structural applications.
BIBLIOGRAPHY


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