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DIRECTIONALLY SOLIDIFIED COMPOSITES: KNOWN ALSO AS 'IN SITU' COMPOSITES, OR DIRECTIONALLY SOLIDIFIED EUTECTICS

NATIONAL MATERIALS ADVISORY BOARD (NAS-NAE)

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Directionally Solidified Composites

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This report, the results of a study by the ad hoc Committee on Directional Solidification of the National Materials Advisory Board, focuses on the potential of this new class of material, the problems to be faced in designing equipment using it, and the steps that should be taken to advance the science and technology of directionally solidified composites. In reviewing completed and current work, the Committee found that interest in this class of material is widespread and early progress has been excellent but that there are still many unknowns and a considerable research and development effort is required before such applications as gas turbine engine blades are warranted.

The Committee concluded that research and development work on alloy systems, protective coatings, joining, and inspection has been insufficient and that mechanical and physical testing has not been conducted under a sufficiently wide range of conditions. The Committee noted that additional alloy systems (e.g., silicides because of their potential for oxidation resistance) as well as off-axis properties, thermal and mechanical stability, section size and geometry effects, and other problem areas are in need of further investigation. Thus a fairly extensive support program is recommended prior to rig and engine testing. Because of the sizeable investment demanded by such a program, periodic reviews of technical status, emerging requirements, and technical and economic feasibility will be required.

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NOTICE

The project which is the subject of this report was approved by the Governing Board of the National Research Council, acting in behalf of the National Academy of Sciences. Such approval reflects the Board's judgment that the project is of national importance and appropriate with respect to both the purposes and resources of the National Research Council.

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United Aircraft Corporation
400 Main Street
East Hartford, Ct 06108

* Resigned Feb. 29, 1972. Now located in Brussels, Belgium.

AD HOC COMMITTEE ON DIRECTIONAL SOLIDIFICATION

Liaison Representatives

Department of Defense

Mr. Jerome Persh
Staff Specialist for Materials and Structures (Engineering Technology)
Office, Director of Defense Research and Engineering
Department of Defense
Washington, D.C. 20301

Department of the Army

Dr. A. E. Gorum
Dr. Paul Ahearn
U. S. Army Materials and Mechanics Research Center
Watertown, Ma 02172

Department of the Navy

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Naval Air Systems Command
Department of the Navy
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Department of the Air Force

Capt. David Rice
Attn: AFML/LLC
Wright-Patterson Air Force Base
Ohio 45433

National Aeronautics and Space Administration

Dr. Richard L. Ashbrock
Mail Stop 49-1
NASA Lewis Research Center
21000 Brookpark Road
Cleveland, Oh 44135

Mr. Joseph Maltz
Code RWM
Materials & Structures Div.
National Aeronautics and Space Administration
Washington, D.C. 20546

NMAB Staff Member

Dr. Joseph R. Lane
Staff Metallurgist
National Materials Advisory Board
NAS-NAE
2101 Constitution Avenue
Washington, D.C. 20418

ABSTRACT

The formation of an *in situ* composite by means of appropriate directional solidification is a process with great potential for high-strength, high-temperature materials. An obvious primary application is for gas turbine blades, but non-structural applications also are possible and at least one of these is already a commercial product.

This report, the results of a study by the ad hoc Committee on Directional Solidification of the National Materials Advisory Board, focuses on the potential of this new class of material, the problems to be faced in designing equipment using it, and the steps that should be taken to advance the science and technology of directionally solidified composites. In reviewing completed and current work, the Committee found that interest in this class of material is widespread and early progress has been excellent but that there are still many unknowns and a considerable research and development effort is required before such applications as gas turbine engine blades are warranted.

The Committee concluded that research and development work on alloy systems, protective coatings, joining, and inspection has been insufficient and that mechanical and physical testing has not been conducted under a sufficiently wide range of conditions. The Committee noted that additional alloy systems (e.g., silicides because of their potential for oxidation resistance) as well as off-axis properties, thermal and mechanical stability, section size and geometry effects, and other problem areas are in need of further investigation.

Thus, a fairly extensive support program is recommended prior to rig and engine testing. Because of the sizeable investment demanded by such a program, periodic reviews of technical status, emerging requirements, and technical and economic feasibility will be required.

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I. INTRODUCTION

Early in the 1960s the idea for a new class of engineering material was conceived. What resulted was the discovery that eutectic alloys can be manufactured to possess unusual, highly anisotropic microstructures and properties. These materials have come to be variously called directionally solidified composites, in situ composites, and directionally solidified eutectics.

The National Academy of Sciences was asked by the Department of Defense to convene a committee to review the state-of-the-art of unidirectional castings, in order to recommend the research and development needed to advance the technology. This report constitutes its findings.

It was already known that when a eutectic alloy (metallic or ceramic) was solidified with little attention to the mode of solidification, the resultant microstructure frequently consisted of two (or more) phases arranged more-or-less randomly. The size and shape of the interlocking crystals exhibited very little pattern or order. Properties were generally isotropic on a macroscopic scale and, since "statistical averaging" frequently means that the least common denominator prevails, the mechanical and physical properties were generally not very exciting. In addition, it had been known since the 1920s that, on a microscopic scale, individual crystallites form from the melt by nucleating and growing parallel to the local freezing direction. In two-phase structures both phases solidify simultaneously into the liquid in a direction parallel to the local direction of heat flow and adjacent to one another.

The new idea, however, prompted investigators to ask: If the solidification process is controlled to produce eutectic ingots in which the microscopic crystals are aligned parallel to one another in a regular array over large distances, will not the ingots have unusual and highly anisotropic microstructures and, hence, unusual and useful new properties?

Thus, efforts were undertaken to try to control the solidification process on the macroscopic scale in such a way that a substantially planar solid-liquid interface would sweep through an ingot specimen from one end to the other. This was accomplished by constructing an apparatus not unlike that used to grow single crystals of pure metals. A common arrangement, still used, is to put an alloy mixture of eutectic composition in an elongated tube or crucible, melt it in a furnace, and slowly withdraw the crucible from the furnace so that freezing of the entire ingot progresses parallel to the axis of the crucible. When this is done the microscopic crystals form in a parallel array which can extend throughout the entire ingot. The individual crystals in such an ingot are often found to be of markedly elongated shape; their length in a direction parallel to the long axis of the crucible can lead to an aspect ratio of 1000:1 to 10,000:1 or even greater.

If unidirectionally solidified ingots of binary eutectics are sectioned transverse to the growth direction, two types of microstructure are commonly observed. One consists of numerous small, nearly circular sections across fibrous crystals of one phase, which are imbedded within the other phase. The second microstructure consists of numerous

interlocking lamellar crystals of both phases. The two kinds of microstructures reveal that directionally solidified eutectic composites often consist of either parallel fiber-like crystals of one phase in a matrix of the other or parallel alternating lamellae of the two phases. More complicated morphologies sometimes occur or can be made to occur, particularly in ternary and more complex alloys. It should be noted, however, that the principle has been applied to monotectic reactions as well as to monovariant eutectic reactions in ternary and other more complex alloy systems.

In the case of both the fibrous and lamellar morphologies the thinnest dimension of the crystals on the transverse section is on the order of microns. Both theory and practice indicate that the mean value of this dimension, or of the interparticle spacing, can be varied somewhat. This characteristic dimension is determined in large part by the solidification rate according to the functional relationship $\lambda^2 R$ = constant (λ is spacing, R is solidification rate). For most potential applications, thinner crystals are thought to be preferable to thicker crystals.

The relationship, $\lambda^2 R$ = constant, however, is valid over only a certain range of R values, restricted by impurity content. The reason for this is that as R is increased (to make thinner crystals) a point is reached at which impurity effects begin to dominate and control the microstructure. When this happens the parallel arrangement of crystallites produced by solidification from a planar freezing front begins to degenerate to a more random array. In practice, parallel phase arrangements can be achieved

with characteristic crystal dimensions (transverse direction) ranging from about 0.1 to 10 microns which correspond to four orders of magnitude in solidification rate.

The arrangement of elongated microcrystals in a directionally solidified composite (DSC) specimen is rarely a perfect parallel arrangement, i.e., the fibrous morphology rarely consists of a perfect geometrical packing of fibers surrounded by a matrix and the lamellar morphology rarely consists of a perfect alternation of crystallites of the two phases. Minor perturbations in the geometrical arrangement of the phase particles, resulting from minor fluctuations in the nucleation and growth processes that occur during solidification, are usually observed. Such irregularities probably have only a minor effect on certain properties of controlled eutectics (e.g., strength), but they can be expected to have a major effect on others (e.g., electrical properties). These structural defects, or terminations as they are called in the technical literature, have been studied extensively because it is believed that a deeper understanding of their origin and development during solidification will lead to further improvement of directionally solidified composites.

Eutectics, by definition, are the lowest melting point alloys for any given set of components. The volumes of the respective phases are fixed by the phase diagram and do not change because of directional solidification. However, preferred crystallographic orientations are often formed during controlled solidification, with the result that the interfaces between the crystallites vary and play a role in property control. In many cases the preferred orientations which develop are associated with low values

of the interfacial energy. Since all the interfaces in a directionally solidified composite tend to have the same crystallographic structure and to be parallel to one another (except in the vicinity of terminations), the vast majority of the interfaces have a low energy. This means that controlled eutectics can be heated to temperatures quite close to the eutectic melting temperature without undergoing structural degradation. Thus, even though eutectics are the lowest melting point alloys in an alloy system, they can be used at much higher homologous temperatures, and it is this feature, among others, which makes them so attractive for applications such as turbine blades.

A. THE PROCESS

To produce directionally solidified composites, suitable arrangements must be made to sweep a substantially planar liquid-solid freezing interface through the casting. Since impurities often cause a macroscopically planar freezing interface to degenerate to a non-planar configuration and the impurity degeneration effect is more pronounced at fast solidification rates or when the thermal gradient in the liquid at the freezing interface is low, an appropriate balance must be achieved. The ratio G/RC_o (where C_o is the impurity concentration, G is the thermal gradient, and R is the solidification rate) must be greater than some critical value to produce a specimen of controlled eutectic. The critical value of G/RC_o varies with the alloy system and is not always known explicitly, but the principle seems to have been demonstrated adequately.

Extensive research efforts have shown that when eutectics are unidirectionally solidified at an appropriately high value of G/RC_o, highly anisotropic arrays of microcrystals

are formed. In every case the long axis of the crystallites is parallel to the heat flow direction of the object being fabricated. It is safe to conclude that almost any eutectic material can be solidified over sufficiently wide rates to produce directional composites.

B. THE PRODUCT

The microstructure of directionally solidified eutectic composites is commonly either fibrous or lamellar. In either case the thin dimensions of the crystals is measured in microns (0.1 to 10 microns) and the long dimension in terms of the dimensions of the object fabricated (i.e., centimeters or inches). Usually a high degree of preferred orientation is observed (Fig. 1). The advantage of the eutectic process and product is that all phases are formed simultaneously from the melt, largely eliminating both separate fabrication and handling problems and interfacial bonding problems associated with the other composite fabrication techniques. However, since the process can be applied only to components that solidify by a eutectic (or monovariant) reaction, directionally solidified bodies cannot be made from arbitrarily selected components. Nevertheless, there are thousands of eutectics and monovariants that have not been explored, suggesting that major advances may be expected from future research and development programs.

1. Mechanical Properties

The performance of advanced aircraft gas turbine engines would be significantly improved by the development of turbine materials with higher temperature and strength

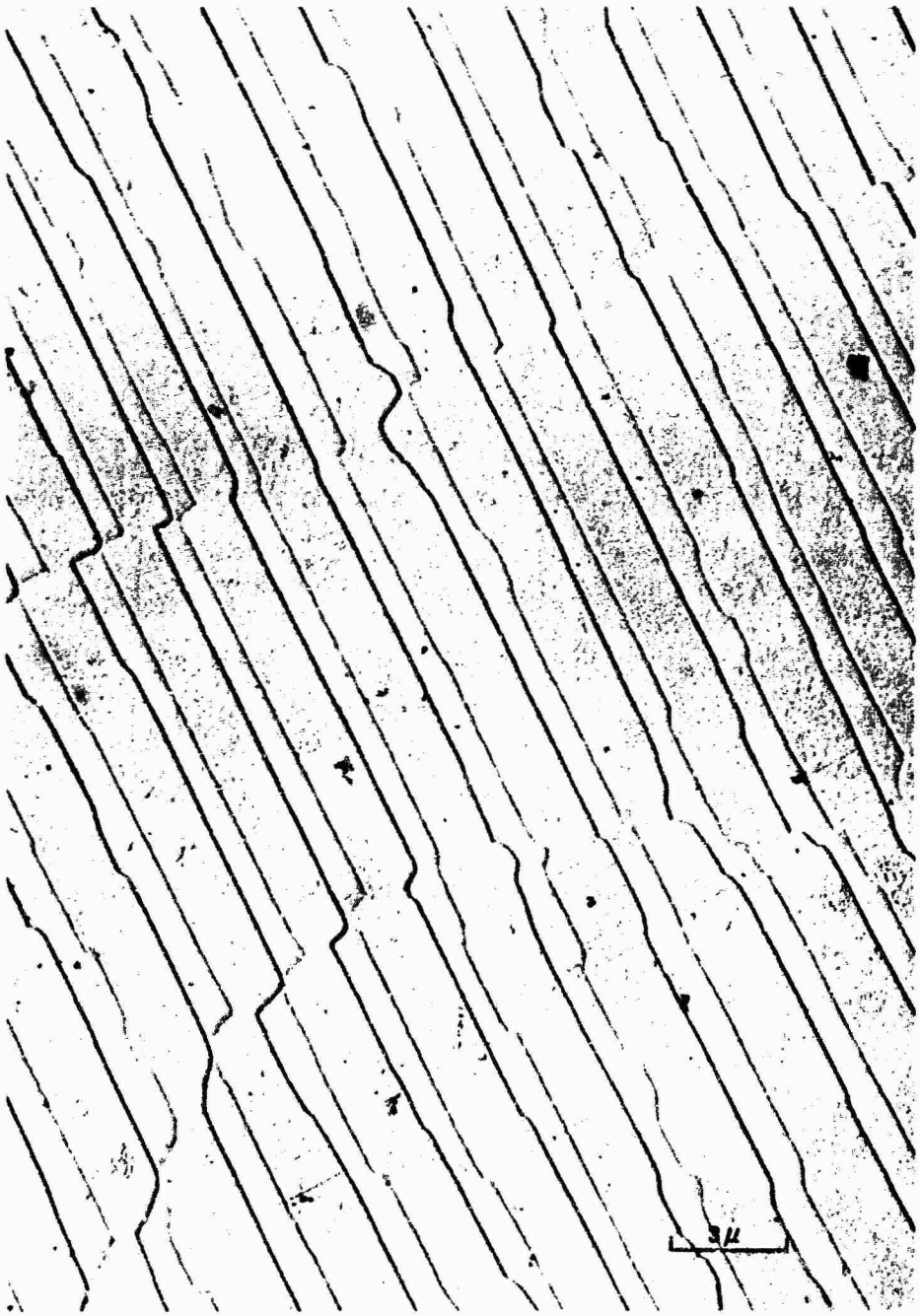

Figure 1. Transverse Microstructure of Off-Eutectic Ni_3Al-Ni_3Nb Composite (Thompson, 1972).

capability. Directionally solidified composites have indicated a potential for use at temperatures at least a 100°F (50°C) higher than is now possible and at stresses that are significantly higher than are permissible with nickel and cobalt-base superalloys. In addition, they possess high-strength values over a wide range of temperatures.

Directionally solidified composites are highly sophisticated materials; they have anisotropic properties and must be grown by highly controlled casting techniques. Advanced and specialized fabrication techniques will probably be required for production of finished components, and improved coatings are needed to take full advantage of the high temperature potential. The successful translation of these materials from the laboratory to an engine will require the coordinated effort of a number of advanced materials, structures, and design groups.

Research and development work in several laboratories (primarily those associated with the aircraft and aerospace industries) has shown that a number of directionally solidified composites demonstrate blade application potential. Figures 2 through 5 (see pp.14-17) illustrate the range of DSC mechanical properties.

2. Non-structural Applications

Eutectics for non-structural applications comprise a much broader and more diverse field than those for structural uses. The diversity of possible non-structural applications of directionally solidified composites is indicated by the set of matrix/inclusion possibilities presented in Table I.

TABLE I

Possible Applications of Non-Structural Eutectics

Inclusion \ Matrix	Conductor	Semiconductor	Insulator
Conductor	Superconductor Magnets Field Emitters Filters Strengthened Electrical Conductors	Magnetic Probes Galvanomagnetic Devices Infrared Dectors Optical Polarizers	Anisotropic Heat Conductors Substrates Capacitors
Semiconductor		Variable Bandgap Semiconductors Optical Polarizers	Optical Devices
Insulator	Strengthened Electrical Conductors Capacitors	Optical Polarizers Acoustic Devices	Optical Devices Magnets

The problems of scale-up to larger sizes and quantities probably would not occur with non-structural applications since tonnage applications are not envisioned. Applications based on physical properties are considered to be a fruitful area for research and development work.

II. ALLOY DEVELOPMENT

The utilization of directionally solidified composites, both as structural and non-structural materials, requires specific property combinations. In the context of its task, the Committee focused attention particularly on structural usage involving temperatures in excess of 2000° F (1093°C); therefore properties sought include high-temperature strength, oxidation resistance, sulfidation resistance, and structural stability. The subject of coatings is inextricably involved.

Alloy development constitutes a critical first step in the selection and promotion of promising alloys. As such, only limited screening data are generated but these are vital to detailed characterization; the latter then serves in preliminary design considerations and also delineates problem areas that may limit application of a particular alloy. Representative screening data, vis-a vis mechanical behavior, and the potential of currently studied composites are presented in Figures 2-5.

A. STATUS

A relatively comprehensive review of the literature was conducted in order to assess the current status of alloy development in directionally solidified composites. Input was received from members of the Committee and a survey was conducted by the Metals and Ceramic Information Center at the Battelle Laboratories, Columbus, Ohio. The review revealed that significant work has been and is being carried out on metal systems, particularly those based on nickel or

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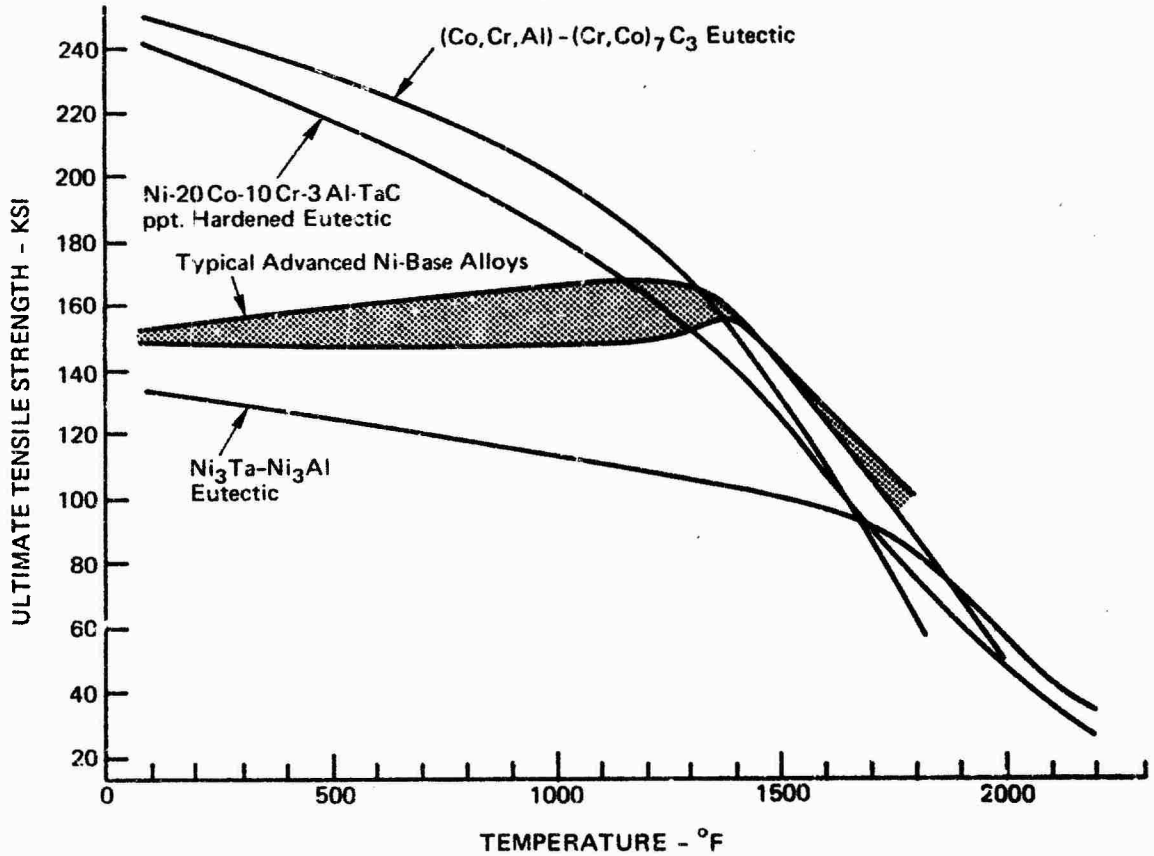

Figure 2. Temperature Dependence of the Longitudinal Tensile Strengths of Fibrous Eutectics (adapted from Thompson, 1972).

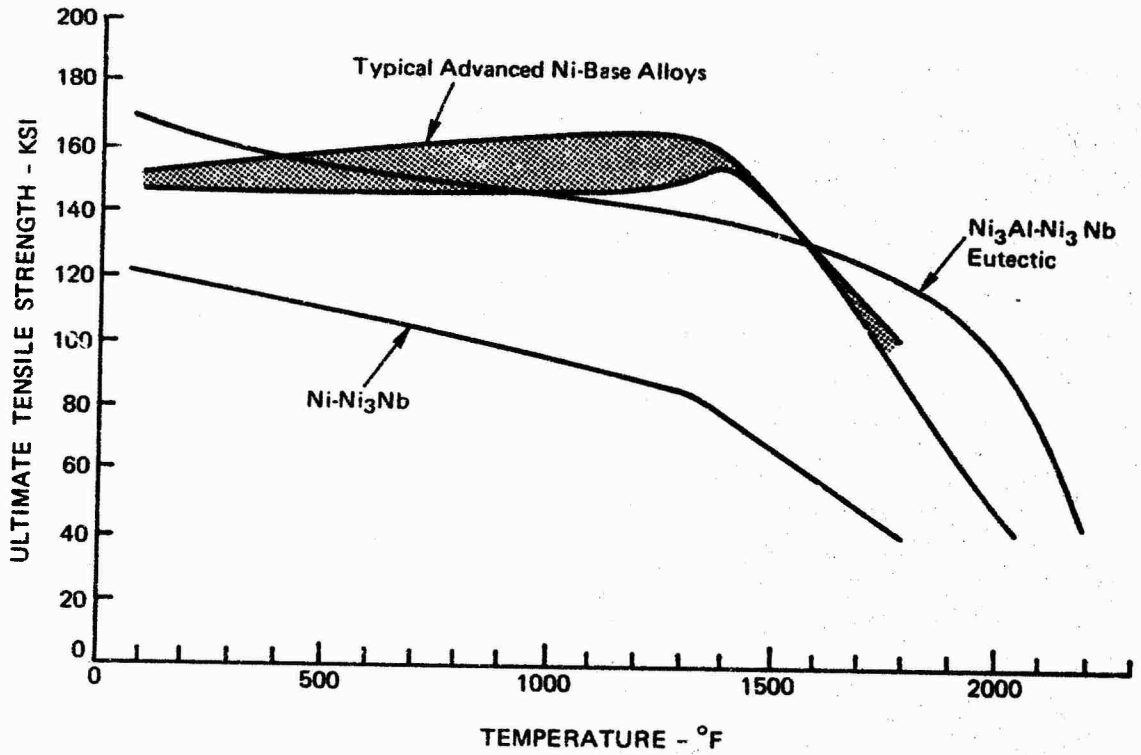

Figure 3 Temperature Dependence of the Longitudinal Tensile Strengths of Lamellar Eutectics Compared to Strongest Nickel-Base Alloy (adapted from Thompson, 1972).

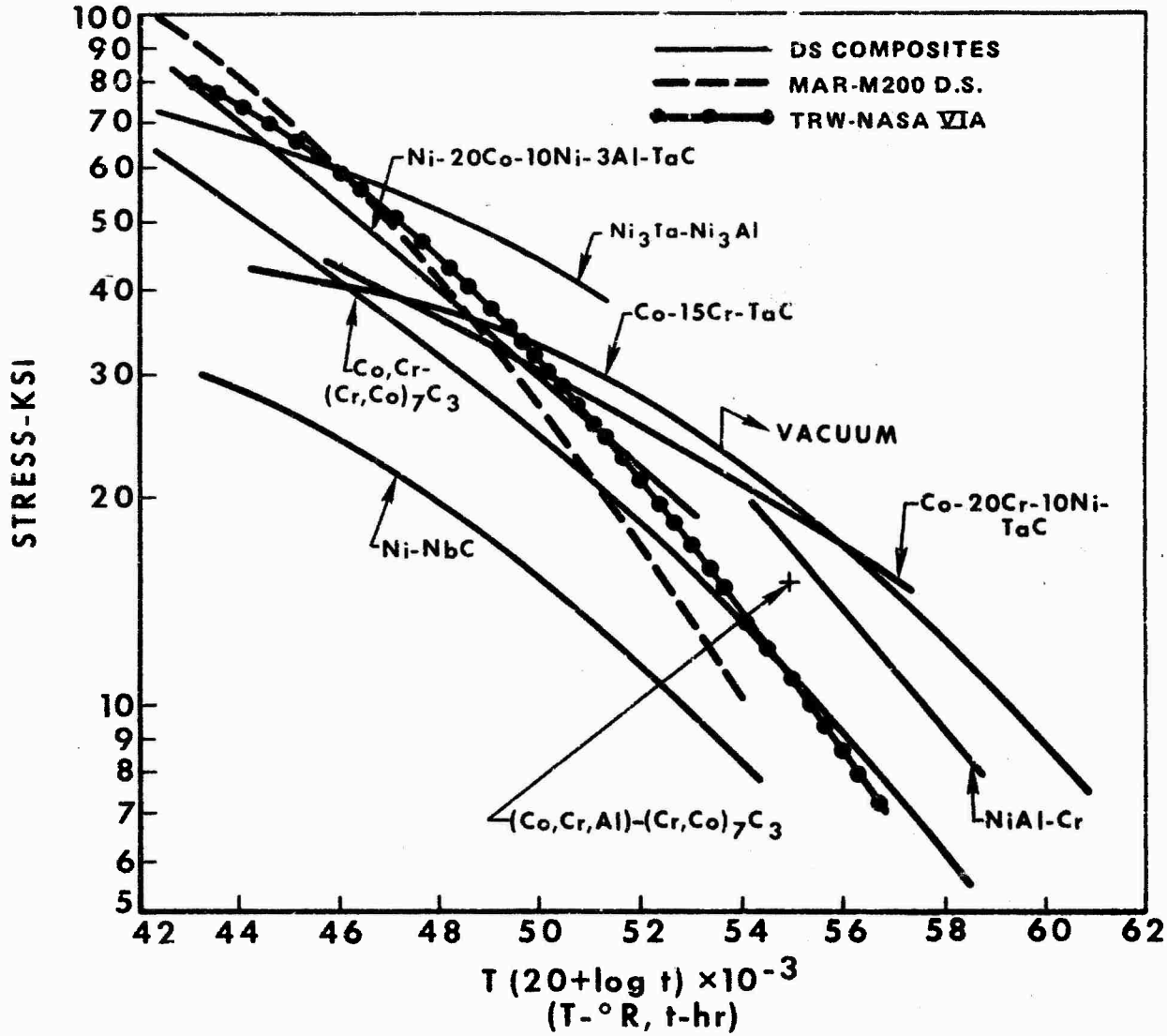

Figure 4 Larson-Miller Parameter Curves for Rupture of Fibrous Eutectics (adapted from Thompson, 1972).

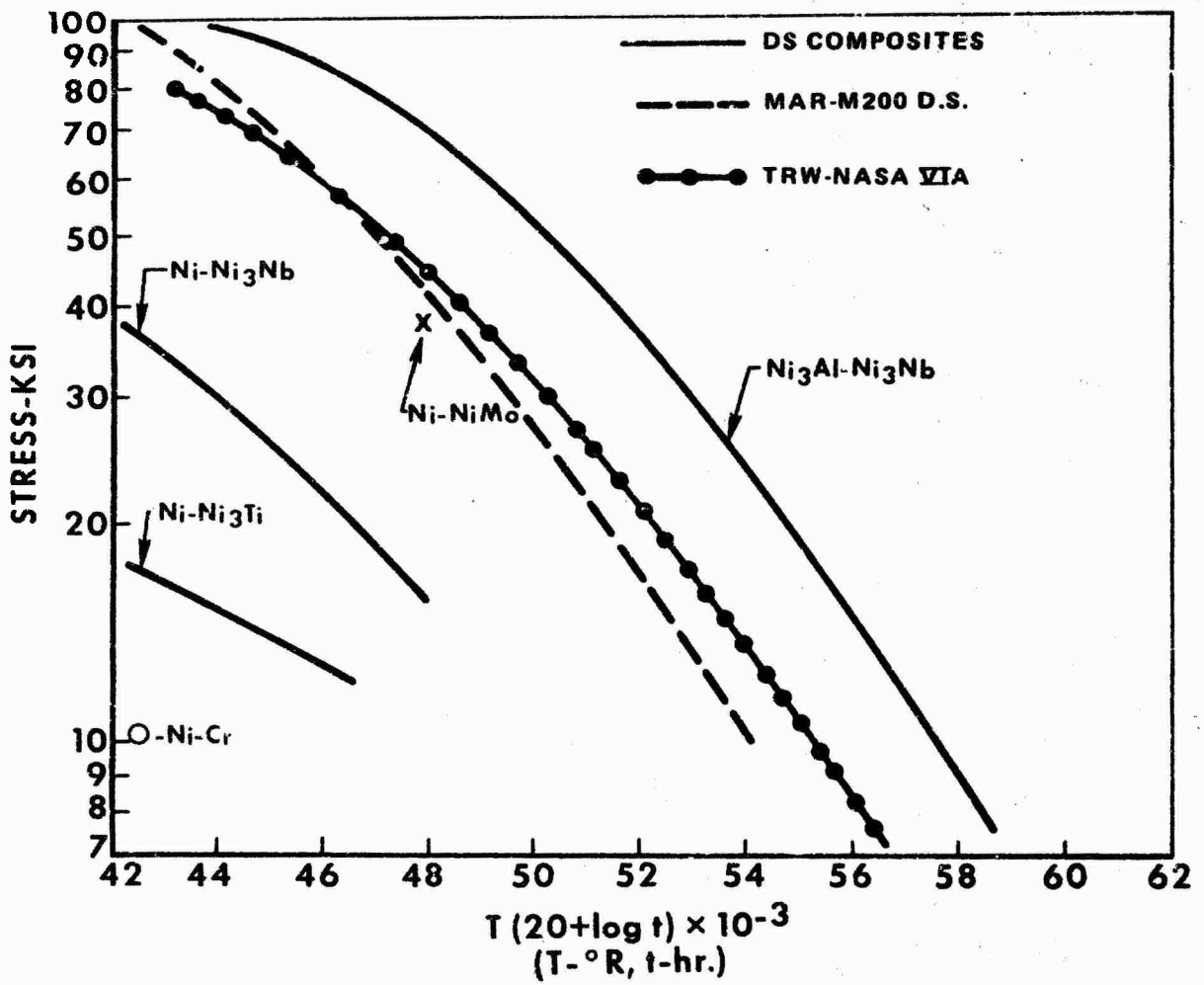

Figure 5 Larson-Miller Parameter Curves for Rupture of Lamellar Eutectics (adapted from Thompson, 1972).

cobalt. Oxide eutectics are relatively unexplored although there are indications of future interest. No information exists in the open literature on silicide systems prepared by directional solidification.

B. CLASSIFICATION

Table II lists the systems for which some reasonable level of understanding of phase-equilibria and/or mechanical behavior has been established. From the standpoint of alloy development, it is convenient to use the following scheme of categorization: metal-metal; metal-intermediate phase; intermetallic-intermetallic; ceramic-metal; ceramic-ceramic.

Strength at high temperatures and corrosion-erosion resistance are the main incentives for considering the use of ceramics in high-performance gas turbines. Oxide in comparison to non-oxide ceramics offer the obvious advantage of oxidation resistance, but as a class they are generally less refractory and more prone to thermal shock than many carbides, nitrides, borides, and silicides. Certain non-oxide ceramics such as silicon carbide and silicon nitride have outstanding corrosion-erosion resistance because of the formation of a passivating layer of silica.

C. POSSIBLE APPROACHES AND GUIDELINES

The formation of aligned composite structures by *in situ* eutectic growth has the advantages of high density, morphologically stable structures at temperatures approaching the eutectic isotherm, integral bonding between fibers and matrix, uniform distribution of phases, and some control over the spacing of the phases. Major problems facing a systematic study of directionally solidified non-oxide ceramic eutectics

TABLE II

Survey and Categorization of Directionally Solidified Composites

System	Alloy	Reference
Metal-Metal	Ni-W	Kurz and Lux, 1971
	Ni-Cr	Kossowsky, 1970
Metal-Intermediate Phase	Cr-NiAl	Cline and Walter, 1970; Cline et al, 1971
	Cr(Mo)-NiAl	Cline and Walter, 1970; Cline et al, 1971
	Cr(W)-NiAl, Cr(V)-NiAl	Cline and Walter, 1970
	Co-CoAl	Cline, 1967
	Ni-Ni_3Nb	Annarum and Turpin, 1972; Hoover and Hertzberg, 1971
	Ni-Ni_3B	Yue et al., 1967
	Co-Co_3Nb	Colling and Kossowsky, 1971; Gudas, 1971
	Ni, Cr-NiBe	Yuan-Shou and Griffiths, 1970
	Co, Cr-$Cr_{7-x}Co_xC_3$	Sahm et al., 1972; Thompson and Lemkey, 1970; Thompson et al., 1970; Koss and Copley, 1971.
	Co, Cr-$Cr_{7-x}Co_xC_3$	Sahm et al., 1972; Thompson et al., 1970; Koss and Copley, 1971
	Ni, Co, Cr+Monocarbides (Ti, V, Zr, Hr, Ta, Nb)	United Aircraft Corp., 1970; Lemkey and Thompson, 1971; Thompson, 1971
	Co-TaC; Co, Cr-TaC; Ni, Cr-TaC	Bibring et al., 1969, 1970, 1971; Livingston, 1972
Intermetallic-Intermetallic	Ni_3Al-Ni_3Nb	Thompson, 1970
Ceramic-Metal	ZrO_2-W	Watson and Chapman, 1972
	UO_2-W	Chapman et al., 1970
	Cr_2O_3-Mo; Cr_2O_3-Re; Cr_2O_3-W; MgO-W	Nelson and Rasmussen, 1970
Ceramic-Ceramic	Al_2O_3-TiO_2	Rowcliffe et al., 1969
	Al_2O-ZrO_2, Al_2O_3-ZrO_2 (Y_2O_3 stabilized)	Schmid and Viechnicki, 1970; Hulse and Blatt, 1971
	ZrO_2-Y_2O_3	Schmid and Viechnicki, 1970; Hulse and Blatt, 1971
	$BaWO_4$-WO_3; SiO, WO_3-WO_3; $BaTiO_3$-Ba_2TiO_4; Fe_2O_3-$LaFe_{12}O_{19}$	Hulse and Blatt, 1971

are the absence of phase equilibria data and the sophistication of the equipment needed to maintain stable liquid-solid interfaces at temperatures $\gtrsim$1093°F ($\gtrsim$ 2000°C).

In Tables III and IV candidate systems for DSC structures are listed based on the following criteria: oxidation resistance, high-temperature strength, and refractoriness M.P. >1093°F (> 2000°C). Mono-carbides are included because of their excellent creep resistance although some have poor corrosion resistance.

The materials listed in Table III are divided into two groups: SiC, Si_3N_4 and AlN, which have relatively low thermal expansion coefficients, and Ta_2B_{17}, Nb_2Be_{17}, $TaSi_2$, $MoSi_2$, WSi_2, ZrB_2, HfB_2, TiC, ZrC, and HfC. Because of thermal expansion mismatch, <u>in situ</u> composites made between members of these two groups are more likely to be weakened by micro-cracking than are composites between members of the same group (excepting the beryllides). Fragmentary data available on interactions between candidate materials are summarized in Table IV. Little information is available on eutectic compositions, even in binary systems. Based on this information and with oxidation resistance, high-temperature strength, and refractoriness as criteria, mixed compounds (e.g., Si_3N_4-AlN and silicide-boride systems) may be worth investigating (Harrison).

For both oxide/oxide and non-oxide/non-oxide ceramic compositions, a systematic review focusing on structure and properties is needed. Basic phase diagram information for oxides, upon which preliminary selections can be made, is fragmentary. <u>It is certain, however, that</u>

TABLE III

Properties of (Non-Oxide Ceramic) Candidate Gas-Turbine Materials*

Material	Melting Point	Density (g/cm^3)	Flexural Strength @ 2370 °F (1300 °C) (psi) x 10^{-3}	Young's Modulus (psi) x 10^{-6} Rm.Temp.	Av. Coeff. of Linear Thermal Expansion - in. per. in./°C x 10^{-6}	Oxidation Resistance
SiC (hot pressed)	5125 °F dec. (2830 °C)	3.2	55	61	5	excellent <2730 °F (<1500 °C)
Si_3N_4 (hot pressed)	3400 °F sub. (1870 °C)	3.2	60	45	3	excellent <2730 °F (<1500 °C)
AlN	4350 °F sub. (2400 °C)	3.3	20	50	5	good <2010 °F (<1100 °C)
Ta_2Be_{17}	3630 °F (2000 °C)	5.1	85	55	15	good <2730 °F (<1500 °C)
Nb_2Be_{17}	3180 °F (1750 °C)	3.3	85	48	16	good <2730 °F (<1500 °C)
$TaSi_2$	3990 °F (2200 °C)	9.1	21		9	excellent <2552 °F (<1400 °C)
$MoSi_2$	3650 °F (2010 °C)	6.3	28	50	8.9	excellent ~<3000 °F (~<1650 °C)
WSi_2	3850 °F (2120 °C)	9.9	58	65	8.4	excellent <3000 °F (<1650 °C)
ZrB_2	5500 °F (3040 °C)	6.1	**	73	8.3	good <2370 °F (<1300 °C)
HfB_2	5880 °F (3250 °C)	11.2	**	70	7.6	good <2370 °F (<1300 °C)
TiC	5560 °F (3070 °C)	4.9	35	62	8.5	fair <1470 °F (< 800 °C)
ZrC	6200 °F (3420 °C)	6.6	30		7.7	fair <1470 °F (< 800 °C)
HfC	7100 °F (3930 °C)	12.[illegible]	30	62	7.1	fair <1470 °F (< 800 °C)

* Source: Goldschmidt, 1967; Hove (ed.), 1965; Lynch (ed.), 1966.

** No values given, high temperature creep strength called excellent.

TABLE IV

Interactions Between Non-Oxide Ceramic Candidate Materials.*

Material System	Interaction	Comments
HfC-ZrC	Complete miscibility	
HfC-TiC	Complete miscibility	
ZrC-TiC	Complete miscibility	
HfB_2-ZrB_2	Complete miscibility	
ZrB_2-ZrC	Eutectic @ 42 m/o ZrC, 5125 °F (2830 °C)	
TiB_2-TiC	Eutectic @ 57 m/o TiC, 4750 °F (2620 °C)	
HfB_2-HfC	Eutectic @ 34 m/o HfC, 5700 °F (3140 °C)	
$TaSi_2$-TaC	Compound formation (MoSiC), T_{MP} = 3760 °F (2070 °C)	Eutectic troughs surround compound
$TaSi_2$-TiC	Partial solubility	Possible eutectic system
$TaSi_2$-ZrB_2	Partial solubility	Possible eutectic system
$TaSi_2$-$MoSi_2$	Partial solubility	Possible eutectic system
$MoSi_2$-ZrB_2	Partial solubility	Possible eutectic system
$MoSi_2$-TiC	Partial solubility	Possible eutectic system

* Source: Hove (ed.), 1965; Geach and Jones, 1955; Rudy, 1969.

as particular systems assume primary importance, more basic phase equilibria data will be required. Hansen (1958) has shown that silicides form in binary systems of silicon with chromium, columbium, magnesium, molybdenum, tantalum, and thorium, titanium, yttrium, and zirconium. Some work has been reported on multicomponent systems of silicon with transition metals (Gladyshevskii, 1962, pp 46-49; Markiv, et al., 1966; Markiv,et al.,1966, pp 1317-1319.). The only example of directional solidification involving a silicide is that of Crossman and Yue (Yue, et. al., 1967 and Crossman and Yue, June 1971, pp 1545-1555) on the eutectic system $Ti-Ti_5Si_3$. In view of the limited applicability of the Hunt-Jackson theory to oxide eutectics (Rowcliffe, et al., 1969, Schmid and Viechnicki, 1970; Hunt and Jackson, 1966, Viechnicki and Schmid, 1969), considerable work remains to be done to establish a sound theoretical basis for understanding eutectic oxide structures.

Finally, attention is drawn to the extensive work in silicides carried out over a long period of time at the Metallwerke Plansee (Compilation of Work on Silicides, 1952-1968.). This provides a detailed background on the chemistry and crystal structures of silicides, the variation of specific volume with composition for a number of silicide mixtures (e.g., $MoSi_2-WSi_2$) and a number of equilibrium diagrams of silicide binary systems (e.g., Mo-Si, W-Si, Ta-Si) and ternary systems (e.g., V-Al-Si, Mo-Al-Si, Mo-W-Si). In particular, the information from the ternary phase diagrams could provide a rationale for selection of promising silicide mixtures vis-a-vis directional solidification.

D. ASSESSMENT

A significant increase in alloy development studies for high-temperature applications should be a major priority for future funding. Alloys having one ductile phase deserve major support because of their similarity to conventional alloys. Research should include an examination of systems utilizing oxides, carbides, nitrides, silicides, and borides, all systems for which phase diagram studies are also particularly needed. Philosophically, the research should range from basic to developmental and scale-up programs with emphasis on phase equilibria, control of structure, and characterization of pertinent mechanical properties. Such studies must examine compositional latitudes for primary constituents, maximum tolerance for trace or impurity elements, and the effects of these on morphology.

With incentives such as strength at high temperatures and good corrosion-erosion resistance, the potential of oxide ceramic systems or those based on mixtures of carbides, nitrides, silicides, and borides should be examined.

Oxide ceramics have an obvious advantage over non-oxide ceramics in oxidation resistance, but as a class they are generally less refractory and more prone to thermal shock than many carbides, borides, and silicides. Silicon carbide and silicon nitride have outstanding corrosion-erosion resistance because of the formation of a passivating layer of silica.

III. INFLUENCE OF GEOMETRY

The properties of directionally solidified eutectic composites depend on the regularity and directionality of the microstructure. Obtaining a uniform, unidirectional microstructure in a cylindrical casting is relatively easy, but it may be difficult to obtain in a complex shape. Three effects of geometry on directionally solidified eutectic microstructures, however, can be expected and may cause irregularities and discontinuities. First, due to the fact that lamellar or rod structures tend to grow parallel to the growth direction, a change in growth direction can produce a discontinuity in the microstructure. Second, a change in geometry often produces a change in growth rate which, in turn, can produce a discontinuity in the microstructure similar to that caused by convection in the liquid phase or growth rate fluctuations due to other causes, such as irregular heat flow. Third, changes in temperature gradient associated with changes in section size or shape can result in a breakdown of the regular microstructure.

A. ORIENTATION EFFECTS

The geometrical effects of orientation changes on directionally solidified eutectics have received little attention. Most of the work that has been done focused on the effects of changing growth direction, which is always encountered to some degree in irregularly shaped castings.

J. D. Hunt (1963), for example, has shown that the structures of several eutectics depended on the direction in which they were grown. Figure 6A shows the experimental

Figure 6 A

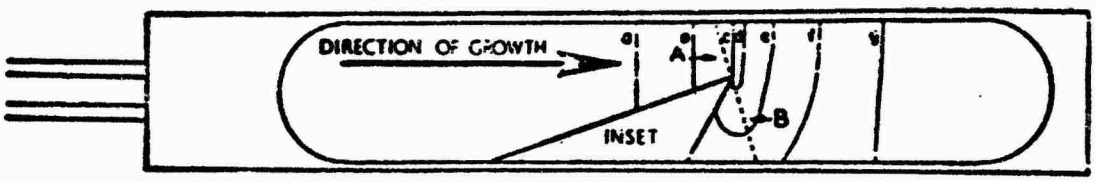

Figure 6B

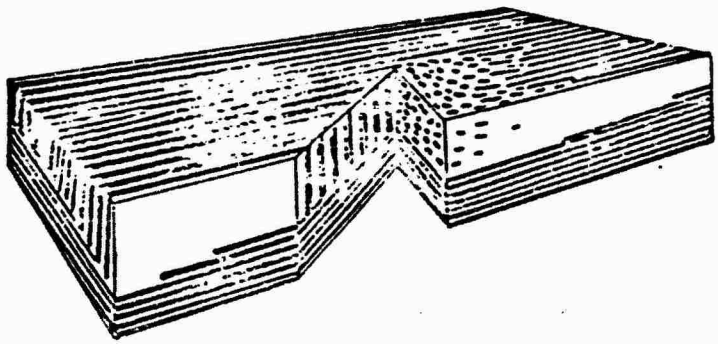

Figure 6 A: A plan view of the boat and graphite inset, showing the shape and position of the solid-liquid interface at successive times (a) to (g);

Figure 6 B A schematic illustration of two grains of a lamellar eutectic, one of which breaks down into a rod-like structure as the grain grows around the inset (Hunt, 1963).

setup used to grow the eutectic around a corner produced by a graphite insert. In growing around the corner, the direction of growth changed; the successive interface positions are shown schematically in Figure 6A. Two distinct cases result and are illustrated in Figure 6B. In case I (the bottom half of the drawing where the lamellae are horizontal), the growth directions both before and during cornering are contained in the lamellar plane. In this case, the lamellae were able to follow around the corner of the inset without disturbance. In case II (the upper half of Figure 6B where the lamellar planes are vertical), the growth direction during cornering is not in the plane of the lamellae. In this case, rod eutectic was found in region B. This applied to Sn-Cd, Pb-Cd, Sn-Zn, and Al-Zn eutectics; for polycrystalline eutectics, those grains which were oriented as in case I were able to grow around the corner without disruption of the structure, whereas for those oriented as in case II, the lamellar structure broke down to rods more or less parallel to the local growth direction. In the Pb-Sn eutectic, a wavy, degenerate lamellar structure was found instead of rods in this region. The Sn-Zn eutectic, which normally grows as a broken lamellar structure, grew as a rod structure during cornering.

Hunt did not investigate the discontinuity in the microstructure at the cornering but was instead interested in the microstructure in the new growth direction. Some of his micrographs indicate a rather sharp discontinuity associated with the change in direction; others, where the change in direction was more gradual, indicate there was a

series of irregularities in the lamellar structure rather than abrupt discontinuity.

It is not possible for a rod eutectic structure to grow around a corner in any orientation without disrupting the structure. Branching of rods or nucleation of new rods in the new growth direction must occur, and either result is bad from a structural point of view since both form potential planes of weakness in the alloy.

The ability of eutectic structures to change direction also has been investigated by Hopkins and Kraft (1968). They studied the development of the preferred growth direction in tin-lead alloys by seeding the eutectic with a single crystal of tin. It was found that the eutectic gradually rotated over several centimeters into its preferred orientation of growth. Similar observations have been made by Van Suchtelen (private communication, 1971) who grew various eutectics with a continuously changing growth direction in a circular boat. The lamellar structure generally followed the local heat flow direction, but there were deviations from it. The eutectic tended to lock in on its preferred growth direction and to rotate out of the preferred direction more slowly than the direction of heat flow was changing.

These observations indicate that the DSC structure can maintain its regularity despite minor changes in growth direction. The importance of these effects in particular alloys will vary and will depend on the degree of perfection of the lamellar structure: the more perfect the lamellar structure, the greater the discontinuity associated with cornering. Thus, a structure containing a large number of lamellar faults should be able to accommodate geometry

changes more readily than a fault-free structure. Recent experiments (Jackson and Miller) conducted with a transparent eutectic system that permitted detailed observation of the cornering indicate that some alloys do not experience great difficulty in growing around corners. The heat flow conditions in these experiments were controlled so that the growth front did not deviate significantly from a plane during the cornering. There, of course, had to be a small leading edge not parallel to the growth front which grew along the surface which was at an angle to the growth front However, it was observed that one of the phases grew as a layer along this surface and that the eutectic structure grew smoothly from it, without observable discontinuity.

These recent experiments indicate that irregularities in DSC structures due to cornering can be minimized by keeping the growth front as plane as possible and by minimizing changes in growth direction due to cornering.

B. GROWTH RATE FLUCTUATIONS

While directionally solidified eutectics are very sensitive to changes in growth rate, the effects of growth rate fluctuations are minimized in alloys of eutectic composition. In off-eutectic alloy compositions, any growth rate fluctuation produces a corresponding fluctuation in the volume fraction of the two phases: one phase gets wider; the other, narrower. If the fluctuation is large enough, the volume fraction of one component goes to zero, and the result is a band of the other component parallel to the interface; all of the lamellae of one phase are terminated.

Figure 7 illustrates how the composition varies across a sample. On the left side of the photograph, the composition is close to the eutectic composition and the effect of the growth rate fluctuation was not very severe; on the right side, the composition was further from the eutectic and the structure was completely disrupted by the growth rate fluctuation. This effect occurs because the liquid at the interface is close to the eutectic composition, and the liquid far from the interface is not (in an off-composition alloy). The boundary layer at the interface associated with this composition change has a thickness that depends on the growth rate. A growth rate fluctuation results in part of this boundary layer being dumped into the solid phase in the form of a change in volume fraction. Irregularities in the microstructure also will result from the buildup of the boundary layer at an increase in section size; there also will be a change due to the partial solidification of the boundary layer where the section size decreased.

Such boundary layers, as noted above, are minimized in alloys of nominal eutectic composition. It is, however, frequently quite difficult to maintain the eutectic composition precisely; in some cases it is even difficult to homogenize the composition of the melt. Therefore, from a practical point of view, these effects are always present to some degree and are likely to be even harder to eliminate from pseudobinary, ternary, and more complex alloys.

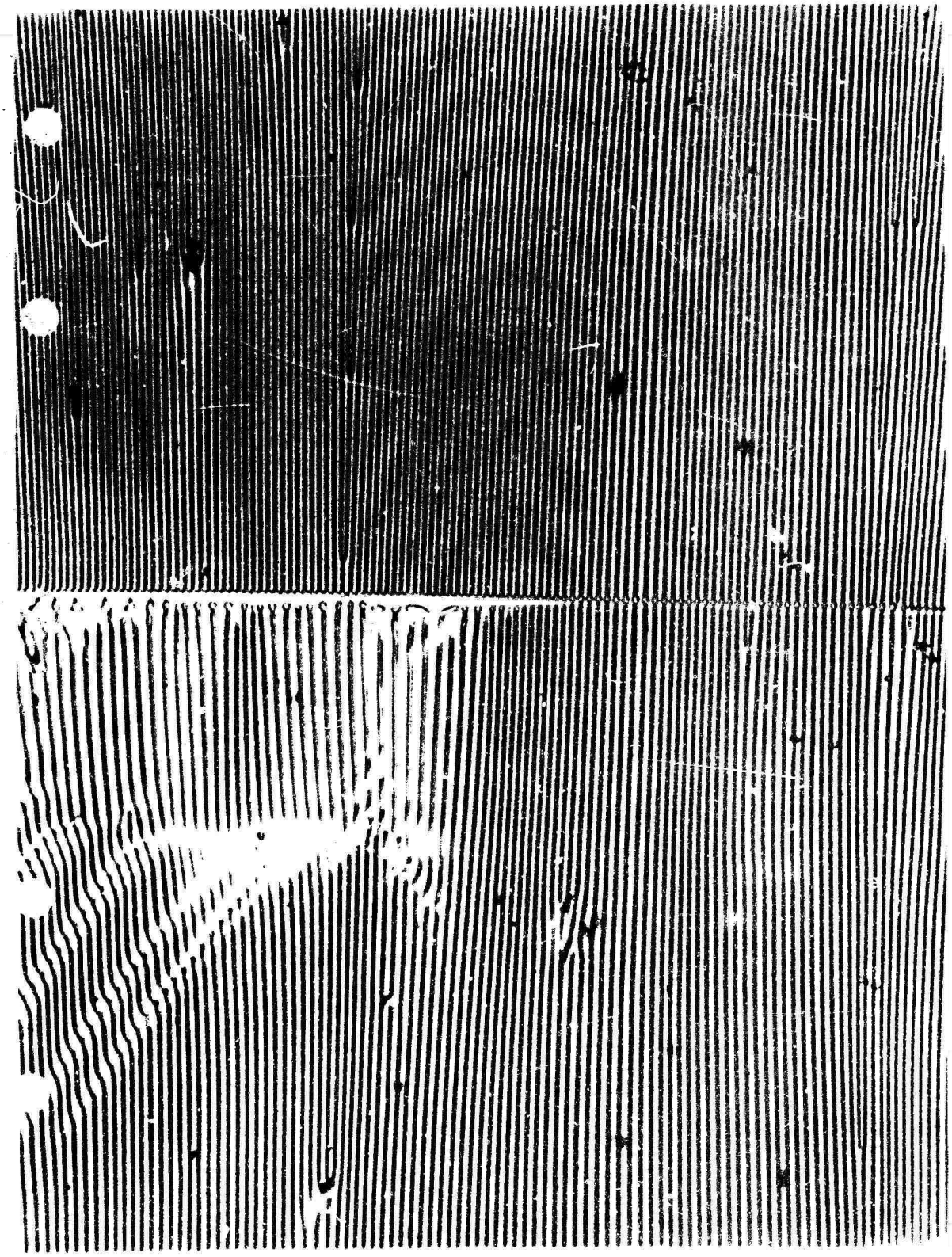

Figure 7. Structural irregularity caused by growth rate fluctuation. Composition of [illegible] left is closer to eutectic composition than that [illegible] the right.

In addition to the boundary layer effects, the lamellar spacing varies with growth rate as:

$$\lambda \propto R^{-1/2},$$

where λ = spacing and R = solidification rate. It usually takes some time for the lamellar spacing to respond to a growth rate change and, by themselves, small amplitude, rapid fluctuations in rate are unlikely to have a significant effect on the microstructure. Of course, there can be effects due to changes in spacing superimposed on the banding due to the boundary layer, which would not be present without the boundary layer. However, a large fluctuation in growth rate will produce a sudden change in lamellar spacing where all the lamellae split or join in pairs. This front could form a plane of weakness in the resulting alloy. The effect of such a change in lamellar spacing and the sensitivity to growth in fluctuations will be enhanced by the presence of the boundary layer effects.

C. COLONY FORMATION

As noted above, impurities can cause a macroscopically planar growth front to degenerate into a non-planar configuration. This results in the formation of a colony structure, which represents a breakdown of the directional lamellar growth. In addition, off-composition growth can result in the formation of dendrites of one of the primary phases. Either of these conditions can be minimized by steep temperature gradients and/or slow growth rates.

The condition for the breakdown* of the planar interface is given approximately by:

$$G < \frac{\Delta T_f R}{D},$$

where G is the temperature gradient, D is the diffusion coefficient in the liquid, R is the growth rate, and ΔT_f is the freezing range of the alloy (i.e., the temperature difference between the solidus and liquidus lines on the phase diagram for the particular alloy composition). ΔT_f is small if the alloy has eutectic composition and if the impurity content is low. Off-eutectic composition alloys have large ΔT_f, and impurities also increase ΔT_f. This equation, although only approximate for eutectic alloys, can be considered a useful guideline. Composition, growth rate, and temperature gradient must be chosen carefully for a particular alloy system in order to avoid the breakdown of the regular structure, as well as to take into account the other restraints on these quantities imposed by consideration of available growth times, experimentally accessible temperature gradients, thermal environment, mold geometry, etc. Even though these parameters are chosen so that the alloy can be solidified as a regular composite in most of the casting, large changes in geometry can cause problems. For example, a large change in section can change the growth rate and the temperature gradient, thereby causing breakdown of the regular structure. The growth conditions must be chosen to allow for these variations, so that the breakdown does not occur under the most extreme conditions encountered in the casting.

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* Compared to p. 5 in which the conditions for stability are stated where a ratio <u>greater than</u> a critical value is indicated, here the conditions for instability involve a ratio <u>less than</u> an indicated value.

Recent experiments (Graham, 1973) have shown that complex shapes of high-temperature directionally solidified composites can be cast. The eutectic structure in the initial part of the casting, near the chill, was quite irregular but settled down to regular lamellar growth within several inches of the chill. In regions of the castings where the section was changing slowly, a regular, aligned structure was produced, although control of the growth parameters was necessary to achieve this result. At large changes in section the lamellar structure became irregular. The lamellae tended to follow the growth direction around corners into a shelf in the casting. Property evaluations will be necessary to determine how serious these irregularities are. Evidence was found for the breakdown of the regular lamellar structure to irregular colony growth resulting from changes in solidification parameters during the growth process.

A number of solid and hollow blades have been cast by an engine manufacturer using both production withdrawal equipment and the liquid metal cooling process (Figures 8 and 9). These are being used to carefully document the microstructure in various locations and to obtain mechanical property data on specimens taken from blades. Initial results on determining the microstructural variations within a Ni, Ni_3Al-Ni_3Nb eutectic blade are given in Figure 10. The most uniform structure is obtained in the airfoil where the G/R ratio is best controlled. Regions of cross-section change, such as blend areas and the platform, exhibit non-uniform structures because of deviations in heat and mass flow.

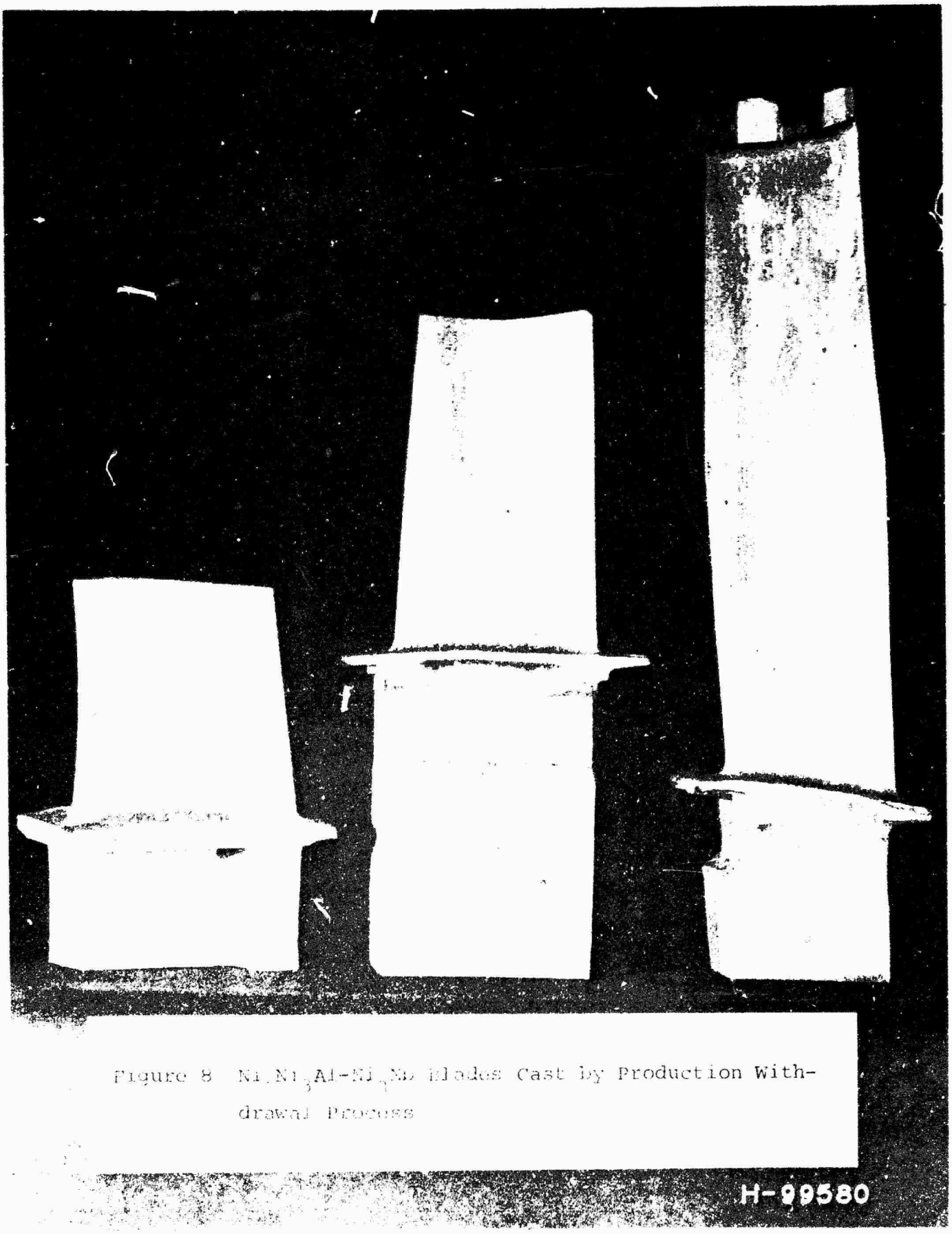


Figure 8 Ni_3Al-Ni_3Nb Blades Cast by Production Withdrawal Process

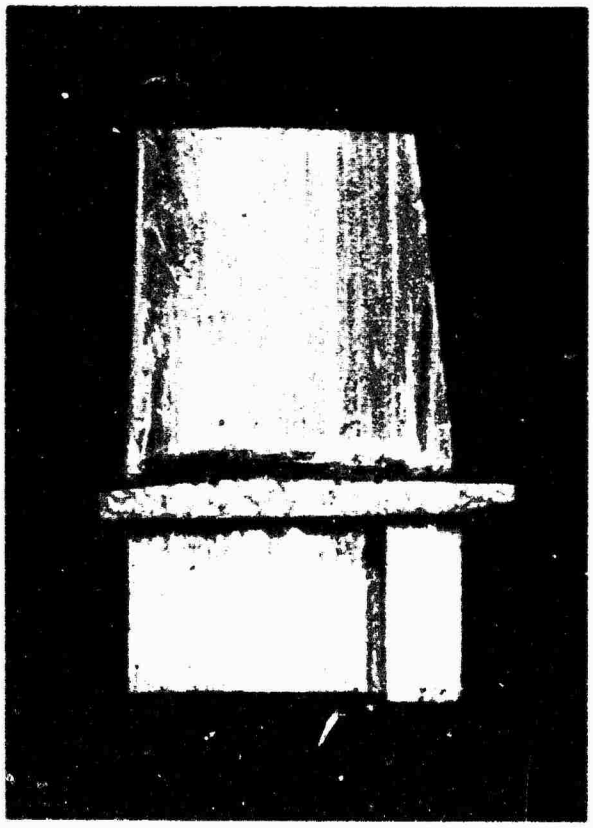
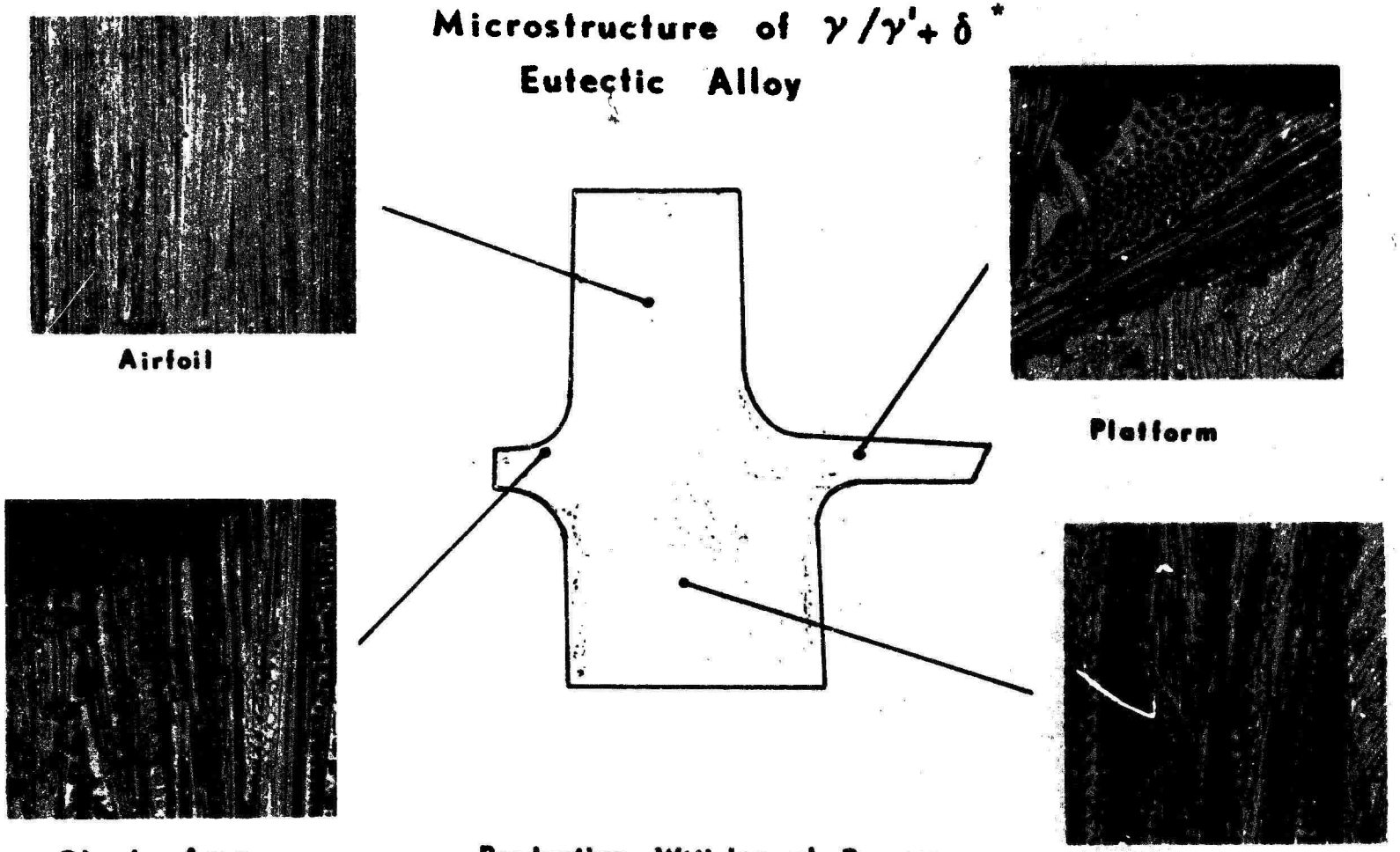


Figure 9 Ni, Ni_3Al-Ni_3Nb Blade Cast by Liquid Metal Cooling Process

Figure 10

Effect of Cross-Section Change on the Microstructure of $\gamma/\gamma' + \delta$ * Eutectic Alloy



Production Withdrawal Process Longitudinal Sections

*Ni, Ni_3Al-Ni_3Nb

D. ASSESSMENT

DSC growth conditions can be controlled to produce castings of complex shapes. While the structure has the desired regularity in regions where the section is changing ing slowly, rapid changes in section produce irregularities in the structure. Experimental work is needed to optimize mold design and solidification conditions to minimize these irregularities.

The extent to which microstructural irregularities adversely affect properties also needs further investigation. For example, having the lamellae change direction into a shelf may be advantageous, but the effects of structural irregularities on mechanical properties, thermal stability, and response to thermal cycling, while not presently known, are not expected to be beneficial.

It is clear that the properties of directionally solidified composites depend to some degree on the regularity and perfection of their microstructure. It is also clear that complex shapes with an ideally aligned structure throughout will be difficult to cast.

Many of the properties of directionally solidified composites can be determined from measurements on regular test specimens cut from carefully directionally solidified ingots. Because of their susceptibility to structural imperfections, however, directionally solidified composites cast in complex shapes are much more likely to have properties that differ from those of test bars than are other structural shapes. To determine the importance of these effects, it will be necessary to undertake tests of parts early in the assessment procedure of new directionally solidified eutectic alloys.

IV. CHARACTERIZATION DATA AND STABILITY

In the alloy development stage only limited screening data are gathered to identify promising alloys. Such data commonly include tensile and creep-rupture data (see Figures 2-5) and very limited amounts of other mechanical property, oxidation, and thermal stability information. Final incorporation of a new material into a gas turbine engine requires extensive design data and, in particular, identification of problem areas that may limit the application of the particular alloy.

Only a few DSC systems have progressed beyond the screening-data stage. Some characterization data (summarized qualitatively in Table V) have been gathered for Ni, Ni_3Al-Ni_3Nb, Ni_3Al-Ni_3Nb, and Co-Tac composites.

Since DSC materials must be considered a new class of materials for gas turbine applications, emphasis at this stage must be on the special features of these materials that may be significantly different from those of the conventional superalloys with which engine designers are familiar. For example, the anisotropy expected in mechanical and physical properties may complicate existing design criteria. The composite nature of these materials could lead to unique damping and thermal-cycling properties. Perhaps most important, new mechanical failure mechanisms could require major alterations in design criteria.

TABLE V

Availability of Characterization Data

	$Ni, Ni_3Al - Ni_3Nb$*	$Ni_3Al - Ni_3Nb$**	Co-TaC***
Creep-rupture	XX	XX	XX
Tensile	XX	XX	XX
Fatigue	X	X	X
Shear	XX	XX	XX
Impact	X	X	XX
Elastic	X	X	
Physical	X	X	X

X - limited data

XX - considerable data

* Thompson and George, 1971 and 1972; Pratt & Whitney Aircraft.

** Thompson, et al., 1969, 1970, 1971, 1972.

*** Bibring, et al., 1972; Benz, et al.

A. CHARACTERIZATION DATA

The major categories of characterization data needed are the following:

1. Creep-rupture -- complete creep curves and stress vs temperature and time for various strains (e.g., 0.1%, 0.2%, 0.5%, and 1%) and for rupture. Creep and rupture strengths are prime criteria for turbine materials, and several DSC systems show high-temperature tension and rupture strengths substantially better than those of current superalloys. However, more work is needed on the mechanisms of creep and rupture in DSC alloys to establish confidence in design criteria. The creep behavior of various superalloys is by now well understood, such that designers can interrelate creep and rupture curves, and can interrelate data at various temperatures and times (e.g., through the Larson-Miller parameter). Similar behavior patterns cannot be assumed for DSC materials until demonstrated.

2. Tensile -- yield strength, UTS, elongation and reduction-of-area vs temperature. Some promising DSC alloys are deficient either in low-temperature ductility or intermediate-temperature strength, and alloy development to remove these deficiences is of high priority. Any properties that make DSC materials inferior to current superalloys will limit their acceptability to engine designers. Low-temperature brittleness may cause the introduction of life-limiting cracks during room-temperature handling and

machining and raise the fear of catastrophic failure of the part during service. Intermediate-temperature weakness may require a major departure in turbine blade design.

3. Fatigue -- thermal, low-cycle, and high-cycle fatigue, including combined-stress tests. Testing should concentrate on the high-temperature range. Low-cycle fatigue from thermal cycling and high-cycle fatigue from vibratory stresses are prominent modes of failure in turbine materials. It is important to assess whether fatigue behavior of DSC candidate materials is qualitatively different from, or similar to, that of currently used superalloys.

4. Shear -- strength vs temperature. Shear tests, not traditionally applied to turbine blade materials will be required, but the aligned interphase interfaces of DSC materials may lead to weakness in shear, which may raise attachment problems.

5. Impact -- ballistic and Charpy V-notch impact strengths at various temperatures. These tests are important to assess the effects of FOD (foreign object damage) on thin airfoil sections. Low impact strength could be a serious limitation for gas turbine engine applications.

6. Elastic -- dynamic elastic modulus vs temperature. Stresses produced by thermal cycling and resonant modes of airfoil should be determined. These stresses and moduli may be highly anisotropic in some DSC materials.

7. Physical -- thermal expansion, thermal conductivity, and specific heat vs temperature; density; incipient melting temperature. Thermal properties influence thermal stresses and cooling efficiency. Increased density would seriously affect disc stresses and weight. A melting temperature too close to the operating temperature is undesirable because of the existence of brief temperature excursions under certain operating conditions.

A major feature of DSC materials is their highly anisotropic microstructure; therefore, a study of the orientation dependence of the mechanical, elastic, and physical properties listed above should receive major emphasis during the characterization data period. A generalized method of characterizing off-axis properties with minimum measurements is desirable.

Data on off-axis properties obtained by the United Aircraft Research Laboratories are presented below (Thompson, 1973):

a. Tension: Table VI below presents the strength of lamellar eutectic Ni_3Al-Ni_3Nb as a function of temperature and orientation.

TABLE VI. Ultimate Strength of Ni_3Al-Ni_3Nb

		Orientation			
Temp.		0°	22.5°	45°	90°
75°F	(24°C)	170,000 psi	105,000	50,000	58,000
1000°F	(540°C)	148,000 psi	130,000	60,000	47,000
1500°F	(815°C)	130,000 psi	120,000	30,000	43,000
2000°F	(1090°C)	95,000 psi	38,000	22,000	30,000

The (Co,Cr)-$(Cr,Co)_7C_3$ eutectic alloy has been studied in tension normal and at 45 degrees to the growth direction at room and elevated temperatures. The strengths of these orientations are plotted as a function of temperature and are compared with the longitudinal orientation in Figure 11. The non-axial strengths are considerably less than that when the fibers are parallel to the stress direction. At temperatures below 1800°F (980°C), the off-axis ductilities are adversely affected by cracks which form in the carbides and propagate along their length.

The tantalum carbide fiber reinforced Co-20Cr-10Ni eutectic alloy has been reported to have the following properties. At room temperature a transverse strength of ≈130 ksi, which is 85% of the longitudinal strength with a tensile elongation of 4.5%, which is 15% of the longitudinal. At 1472°F (800°C), the transverse strength is ≈50 ksi (50% of the longitudinal) with a transverse elongation of ≈7%, which is similar to that longitudinally.

b. Creep Rupture: Off-axis creep properties on the $(Cr,Co)_7C_3$ fiber reinforced (Co,Cr) eutectic alloy in the transverse orientation yielded the following results. The stresses to cause rupture in 100 hours were found to be approxi-

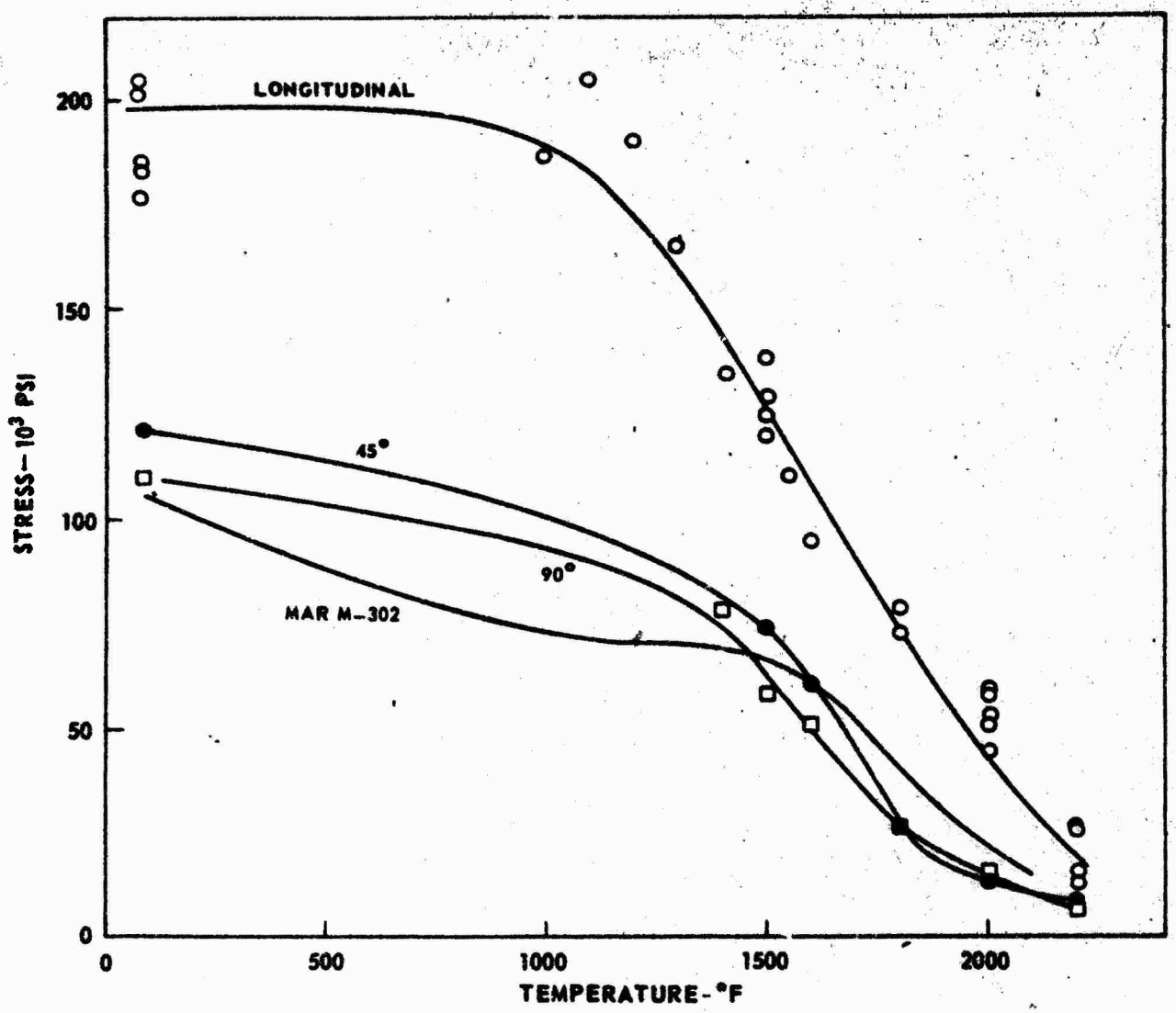


Figure 11 The Tensile Strengths of 73C Longitudinal, Transverse, 45°, compared to MAR M-302.

mately 24, 15, 10, and 4.5 ksi at 1400, 1600, 1800, and 2000°F (760, 870, 980, and 1090°C) respectively. The comparable longitudinal 100 hour rupture strengths are 72, 44, 25, and 13 ksi. The transverse elongations to failure at these temperatures were about 20%.

c. Fatigue: The transversely oriented tantalum carbide fiber reinforced Co-Cr-Ni alloy has been evaluated at room temperature and 1472°F (800°C). The transverse room temperature endurance limit of approximately 65 ksi was 70% of the longitudinal orientation; the 1472°F (800°C) fatigue endurance limit of approximately 36 ksi was 50% of the longitudinal.

Another feature of DSC materials is their sensitivity to processing variables such as solidification rate and temperature gradient. These variables must be well-documented for each test sample and should be in the range achievable in a reasonable commercial process. Eventually, in situ properties must be measured to assure designers that the properties in complex castings or machined parts are equivalent to those in simpler test samples.

Most DSC materials probably will require coatings, which may influence properties. As soon as possible data should be compiled on coated samples.

B. STABILITY

No matter how good its initial properties, an alloy may be rendered useless by structural and property changes that occur in the demanding total environment of a gas turbine engine. Thus, an essential part of the characterization-data stage is a testing of alloy stability (i.e., its resistance to property change during actual or simulated engine operation). The general field of stability of DSC materials not only requires more extensive engineering tests but is a very promising area for fundamental research. Full study may require several different types of tests, including the following:

1. Isothermal: Numerous investigations have shown that the aligned lamellar and rod microstructures of DSC materials are very stable and can sustain prolonged exposure to temperatures above 90 percent of the eutectic temperature with little change in morphology (Salkind, 1969). However, some structural coarsening does occur, particularly for fine regions and for non-faceted rods (Cline, 1971). More research is needed to establish the fundamental mechanisms of coarsening and, in candidate high-temperature systems, the effects of coarsening on mechanical and physical properties.

2. Thermal cycling: There has been comparatively little study of the effects of thermal cycling on the structure and properties of DSC materials. Such cycling may accelerate structural and property changes, particularly in alloy systems with sensitive temperature-dependent phase compositions or for

large thermal expansion mismatches. Temperature gradients may have significant effects on microstructural stability. Impurity effects may play an important role.

Thermal fatigue resistance is commonly tested in fluidized bed or burner rig tests that attempt to simulate the temperature and strain cycles experienced by turbine blades in cyclic engine operation. The total strain experienced includes both "externally-applied" strains produced by temperature gradients and "internally-induced" strains produced by phase changes and expansion mismatches. The latter can be separately investigated by slow thermal cycling tests in which temperature gradients are minimized. External strain can then be added by simple dead loading or by complex thermomechanical fatigue tests, usually on thin-walled tubes, in which any strain-temperature history can be applied to the specimen.

C. ASSESSMENT

More data and an improved understanding of off-axis, mechanical, and physical properties are necessary. Because of the sensitivity of DSC properties to processing variables and surface conditions, characterization data should be extended as soon as possible for each alloy to properties of coated samples. Since all else depends on retention of the original microstructure, studies of microstructural stability should be conducted and should focus on the effects of thermal cycling, temperature gradients, plastic strain, and impurity level.

V. MATERIALS DESIGN INFORMATION (From the viewpoint of the Gas Turbine Producer)

To optimize turbine airfoil parts design, the gas turbine designer requires the various kinds of information listed in Table VII in four groups. Group I information is generally descriptive except for composition limits which are specific. Group II consists of most of the engineering property data required (to give perspective to the amount and kinds of data required, this information has been further detailed in the table). Group III information, while necessary to parts production, is often qualitative, comparative, and descriptive except for impact and thermal fatigue which are specific data. Group IV contains additional information requirements for directionally solidified materials and is largely specific data.

Although all of this information is required for an optimized turbine airfoil parts design, the acquisition of the information proceeds in phases which reflect an increasing commitment to the particular alloy. The phases can be categorized as: preliminary design data; design data minimums; and production design data.

The basis for the acquisition of preliminary design data is that the subject alloy be in the developmental phase (definitely beyond the research stage) and have a reasonably characterized composition, casting process, and heat treatment. If a protective coating is required, a compatible coating/heat treatment should be fairly well defined. Laboratory test data should be sufficient to define the nominal shape and level of each curve (see Table VIII). The preliminary

TABLE VII

DESIGN INFORMATION REQUIREMENTS

I.	Chemical Composition and Composition Control
	Melting and Casting Characteristics
	Form
	Condition
II.	Mechanical Properties
	Elastic Properties
	Physical Properties
	Chemical Properties
	Heat Treatment
III.	Coating Requirements and Characteristics
	Joining Characteristics
	Metallurgical Stability
	Machinability
	Impact Resistance
	Thermal Fatigue
IV.	Off-axis Mechanical Properties
	Transverse Mechanical Properties
	Phase Compatability
	Inspectability

TABLE VIII

DESIGN DATA DETAIL

Mechanical Properties - over temperature range of interest

- Tensile - stress and elongation
- Stress-Rupture
- 0.5% and 1% Creep
- Fatigue
  - HCF - Stress vs Temp. (rotating beam)
  - LCF - Total Strain Range vs Cycles

Elastic Properties - over temperature range

- Dynamic Elastic Modulus
- Poisson's Ratio

Physical Properties -

- Mean Coefficient of Linear Expansion
- Thermal Conductivity
- Specific Heat
- Electrical Resistivity
- Density
- Melting Point (Incipient)

design data evolved at this stage are predictions of the alloy's eventual production capabilities and are based on the combination of engineering information and production experience with similar alloys.

Directionally solidified composites are highly sophisticated materials; they have anisotropic properties; they must be grown by advanced casting techniques; and they possess an almost unique mechanical property-structure relationship. As a consequence, the determination of in situ properties of prototype hardware will be essential to establish preliminary design data. So few data of this kind are available in the literature as to constitute an absence of anything that could be classified as "design data."

For the acquisition of design data minimums, the subject alloys should have reached production status and have an established composition, casting process, and heat treatment as well as a compatible coating heat/treatment if a coating is required. The engineering data should be sufficient to allow statistical definition of tensile, stress rupture, and creep properties and a conservative definition of physical, elastic, and fatigue properties.

For the third and final category, production design data, the engineering data should be sufficient to identify significant property variations due to manufacturing processes and/or engine operation. The data should be sufficient to define the population distribution as affected by any variation so that appropriate modifications can be applied to the base line minimum data.

Since a significant dollar investment is involved in acquiring these data, a step-wise approach is mandatory. The acquisition of this information for a new class of materials will obviously be more complex than for familiar alloy systems.

VI. OXIDATION AND COATINGS

A. OXIDATION

A preliminary evaluation of the oxidation resistance of a number of directionally solidified eutectic alloys has been conducted. An examination of the cyclic oxidation behavior of Ni,Ni_3Al-Ni_3Nb and Co-TaC-111 (Co-10Ni-20Cr-13 wt% TaC) (Bibring et al., 1969, 1970, 1971) at 1400 and 1830 °F (760 and 1000 °C) shows that both materials have about the same oxidation resistance. The oxidation resistance values are somewhat poorer than those for existing nickel-base superalloys and this indicates that both systems will require airfoil and root attachment coatings. Extrapolation of the behavior of various coatings on superalloys is made considerably more uncertain because most available data are in the 1800 °F (980 °C) region, while directionally solidified composites would be used at 1900 to 1950 °F (1040 to 1065 °C). Diffusion and oxidation characteristics can be expected to change at the higher temperatures, and many 1800 °F (980 °C) possibilities would be out of consideration at 1950 °F (1065 °C).

Figure 12 shows the weight gain with time in cyclic oxidation at 1830 °F (1065 °C) for the two eutectic systems, and the corresponding metallographic sections showing oxide penetration are given in Figure 13. At this temperature, the Ni,Ni_3Al-Ni_3Nb forms a surface oxide scale under which a zone denuded of the Ni_3Nb phase is formed. Because the denuded zone separates the oxide from the Ni_3Nb, there is no preferential attack of the Ni_3Nb. This denuded zone

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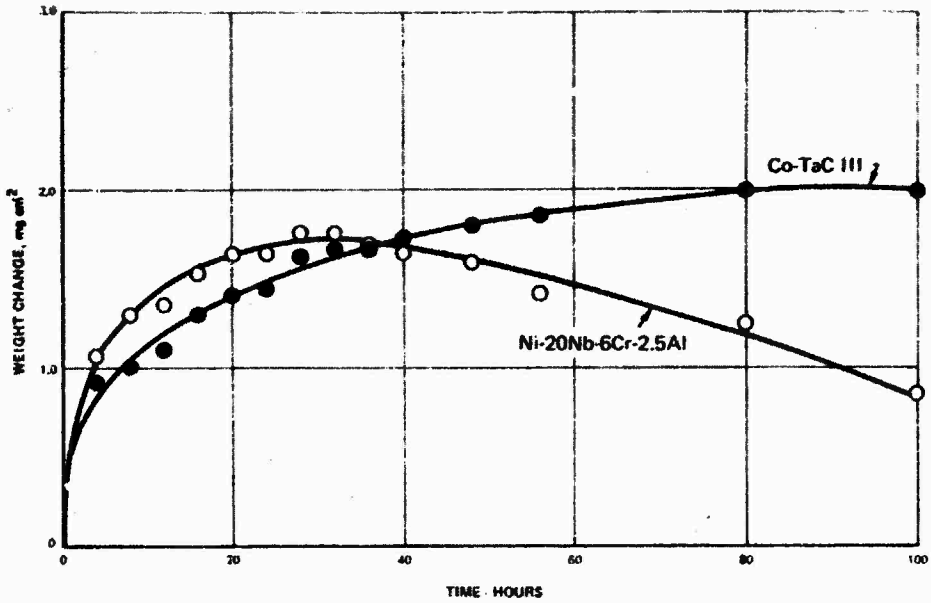

Figure 12 Cyclic Oxidation Behavior of Ni-20Nb-6Cr-2.5Al and Co-TaC at 1830°F (1000°C) in Air.

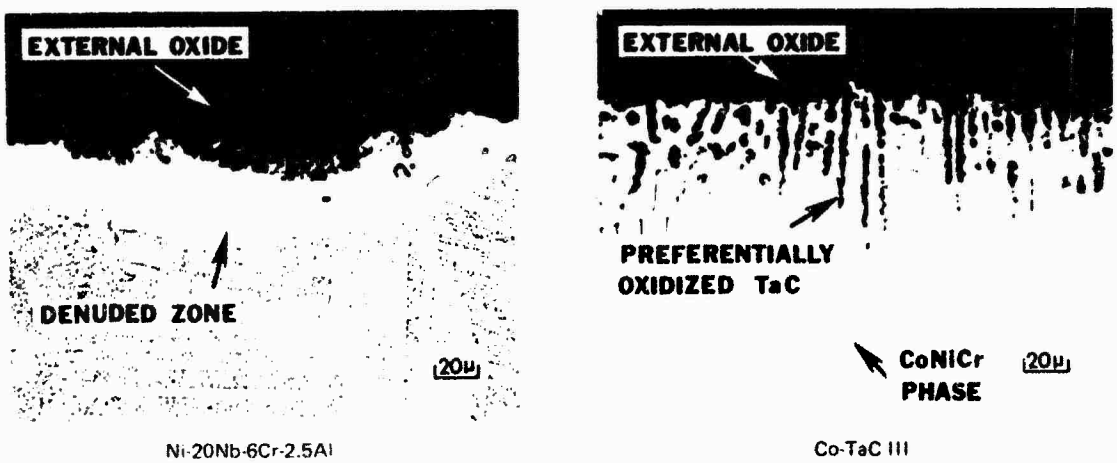

Figure 13 Representative Sections of the Ni-20Nb-6Cr-2.5Al and Co-Tac Alloys after Cyclic Oxidation for 100 hours at 1830°F (1000°C) in Air.

does not form in Co-Tac and, as a result, the TaC fibers are preferentially-oxidized.

Figure 14 shows the weight gain with time for the two eutectic systems at 1400°F (760°C). The weight gain is lower for the Co-TaC. The reason for this can be seen in the metallographic sections of Figure 15. Both the δ and the TaC phases are preferentially attacked. With somewhat greater penetration and a larger volume fraction of δ, the weight gain for Ni,Ni_3Al-Ni_3Nb is greater. In the Co-TaC eutectic, where massive carbides intersect the surface, tantalum oxide is extruded out of the original metal-oxide interface (Figure 16).

It is apparent when the weight change and oxide penetration measurements are compared with existing nickel-base superalloys that the oxidation resistance values of the eutectic composites are somewhat less than those of the chromium oxide forming superalloys such as IN-792 and IN-738 and considerably less than those of aluminum oxide forming superalloys such as B-1900. Airfoil coatings will therefore be required. Based on the 1400°F (760°C) oxidation tests, root coatings also will be needed. Root coatings for hot corrosion protection of existing blade alloys are becoming more common.

The oxidation resistance of Ni_3Al-Ni_3Nb is poorer than that for alloys Ni,Ni_3Al-Ni_3Nb and Co-Tac. For example, at 1800°F (980°C) in cyclic oxidation, a specimen of Ni_3Nb gained 74 mg cm^{-2} in 170 hours while a specimen of pure nickel gained 8 mg cm^{-2} (Thompson, et al.,1969, 1970, 1971; Felten, 1970). Specimens of this eutectic would be slightly

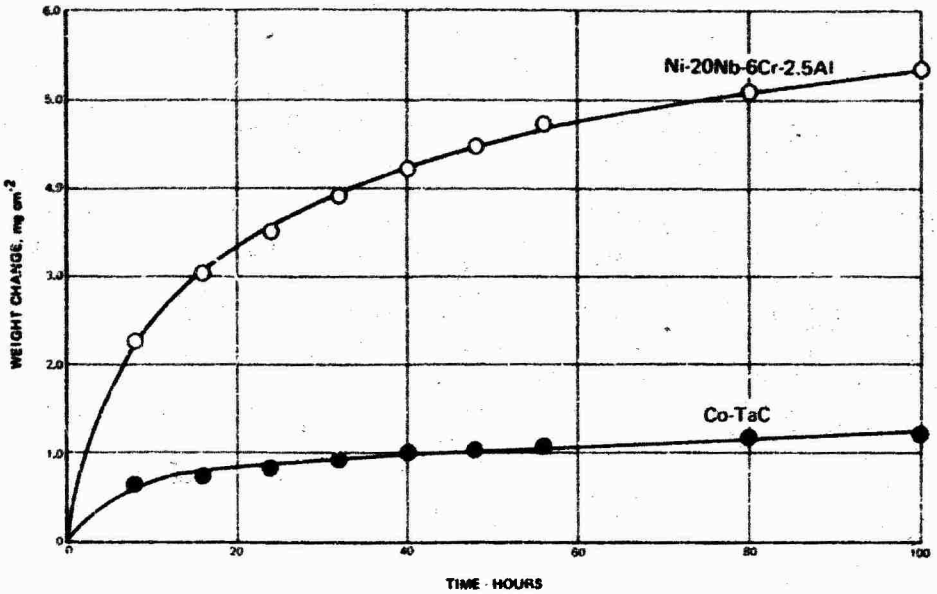

Figure 14 Cyclic Oxidation Behavior of Ni-20Nb-6Cr-2.5Al (δ) and Co-TaC at 1400°F (760°C) in Air.

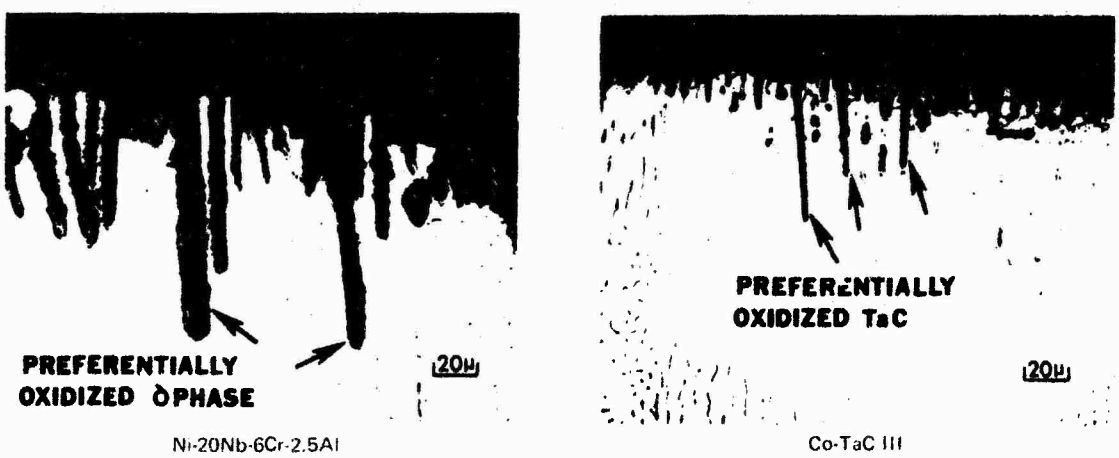

Figure 15 Representative Sections of the Ni-20Nb-6Cr-2.5Al (δ) and Co-TaC Alloys after Cyclic Oxidation for 100 hours at 1400°F (760°C) in Air.

Figure 16 Extrusion of Tantalum Oxide from CoTaC Due to Preferential Oxic ion of Massive TaC Particles at 1400°F (760°C).

less oxidation resistant than pure nickel at this temperature and would be inferior to most contemporary nickel-base superalloys. The primary surface scale formed on the eutectic alloy is nickel oxide (NiO), along with a rather extensive sub-scale of trirutile ($NiNb_2O_6$), spinel ($NiAl_2O_4$), and alumina (Al_2O_3). The oxide is adherent and protective at the lower temperatures but tends to spall at 2000°F (1090°C) and above. The formation of the duplex oxide scale results in the consumption of an excessive amount of metal.

Preliminary hot sulfidation studies have been performed in the laboratory by oxidizing specimens coated with sodium sulfate (Thompson, et al., 1970). The presence of the sulfate decreased the resistance of the eutectic to oxidation at 1562°F (850°C) although not to the degree that is observed in similar tests with nickel-base superalloys of low-chromium content such as B-1900.

B. COATINGS

To fully utilize the high-temperature strength characteristics of directionally solidified eutectics, a coating must be provided. Although overlay coatings of the CoCrAlY type would be expected to provide reasonable protection, it is anticipated that because of the high expected use temperatures of these alloy systems excessive coating-substrate interdiffusion would occur. It would appear that the most efficient coating will be an overlay type based on the NiCrAlY system. An additional factor which must be anticipated and provided for in the practical application of an overlay coating to a turbine blade is

that methods normally used to vapor deposit these coatings are essentially line-of-sight processes which are incapable of coating internal cooling passages or film cooling hole surfaces. It is probable that internal surface coating requirements will be less stringent than those for external airfoil protection. Therefore, diffusion aluminide coatings, particularly those of the outward-diffusion type, could prove adequate. Methods will have to be developed, however, to adequately coat these internal surfaces and to properly blend the two coating systems where they must necessarily overlap while still providing the required total protection for all blade surfaces.

Although root attachment designs and temperatures cannot be accurately assessed at this time, it is probable that the root will also require a coating. This requirement could present a particularly difficult coating problem because of the complex stresses involved in this area.

In general, it must be concluded that alloy and coating development work for improved oxidation resistance is required to provide fully protected DSC airfoils for advanced engine applications.

C. ASSESSMENT

Since DSC systems are intended to be employed at temperatures higher than customary, protective coatings will be required, since most DSC systems have oxidation resistance somewhat poorer than that of commercial alloys. The oxidation behavior of DSC materials is a necessary and important area of research. However, the effort should

focus primarily on coatings.

Coating development is complicated by three factors:

1. The coating must adhere (during application and through thermal cycling) to both constituents of the composite.

2. The types of coatings desired may be difficult to deposit in internal cooling passages.

3. The principal incentive to the use of directionally solidified composites is to permit a higher operating temperature. Coatings such as those now used on blades may be completely unsuitable at a higher temperature level, and a major new development effort may be required.

VII. JOINING

The eutectic alloys of current importance use nickel or cobalt alloy matrices strengthened with rod-like, stable carbides or aligned mixtures of phases such as γ' (Ni_3Al), δ (Ni_3Nb), and γ nickel-rich solid solution. However, their very characteristics pose problems to be overcome prior to their utilization as economical, reliable turbine components. A major concern is that root/disc attachment by conventional mechanical "fir tree" means may be precluded due to the shear characteristics of the axial laminated microstructure. Thus, effective use of eutectics could require the construction of blades by the joining of readily castable segments or the joining of dissimilar conventional materials to the eutectics. To the extent to which parts will not be able to be made to shape by casting increases the urgency of having a joining method developed.

Applicable eutectic material joining methods will be limited due to the effects they have on the composite. For instance, fusion welding methods such as gas, tungsten arc, or electron beam welding would undoubtedly be excluded due to unfavorable results produced by remelting the parent alloy, as well as from thermally induced strain effects. Other joining methods such as upset or flash butt welding requiring significant and abrupt deformations will also be difficult to control so that they are detrimental to the character of the aligned structures. It is expected that methods such as diffusion welding, friction welding, hot isostatic pressure bonding, or brazing would be capable of producing defect-free, structurally sound joints with

the eutectic alloy materials. Some efforts may be warranted on *in situ* bi-casting of materials onto eutectics, although such techniques are difficult to project since reproducible and economical manufacturing methods need development.

ASSESSMENT

To date, very little joining work with eutectic materials has been reported. Some limited studies are in progress, but the state of the art must be considered to be quite underdeveloped. The work underway by engine companies tends to be considered proprietary, making an accurate assessment difficult. However, it will be necessary for more work to be done to establish useful data on the ability to produce sound joints between relevant eutectic systems and other heat-resistant alloys. Data on joining methods, properties, stability, corrosion resistance, reproducibility, and cost are all required.

VIII. CONCEPTUAL DESIGN OF GAS TURBINE ENGINE COMPONENTS

The conceptual design process for evaluating directionally solidified composites for gas turbine component applications consists of:

- Selection of advanced gas turbine engine application(s).
- Establishment of specific engine mission(s).
- Selection of optimized thermodynamic cycles based upon a 100, 150, and 200°F (50, 85 and 93°C) increase in metal operating temperatures.
- Identification of likely turbine components.
- "Design" of selected components with existing information.
- Identification of information yet needed to "design" components.
- Estimation of cost and performance improvement accrued through use of "designed" DSC components.
- Establishment of the worth of DSC components based upon cost and performance trade-off analyses.
- Recommendation of R&D programs as identified in design, cost, and performance trade-off studies.

The extent of material properties information needed by gas turbine engine designers depends upon the commitment, with more detailed and substantiated data being required as consideration progresses from preliminary conceptual design to use in production engines. Component data requirements have evolved through experience and represent information directly related to, or at least correlated to, life expectancy, with some type of failure terminating the usefulness of the

component. These requirements, however, can be applied only to new materials when their modes of failure are similar to those of alloys from which the design criteria experiences have evolved. Thus, one of the principal deterrents encountered in introducing new materials into gas turbine structures is the difficulty of determining the applicability of conventional design criteria. Can the useful life be predicted employing the currently available tools or does the new material exhibit behavior which requires a new approach to design? If new and unproven techniques (i.e., life yet to be established by engine running) are required, material reliability must be established by some sort of prototype evaluation in experimental rigs before commitment to engine tests (failures of components in engines are expensive).

As with all new materials the seriousness of considering directionally solidified composites becomes a function of an applications payoff analysis. This requirement is of primary importance since the optimal design of any gas turbine is an interrelated function of specific mission requirements and available aerodynamics, structural design, and materials technologies. The benefits of directionally solidified composites cannot be realistically appraised (i.e., sufficient to commit them to trial) until they are fitted into a conceptual engine design with a specific mission and the performance assessed against cost.

Besides appraisal, the conceptual design process supplies the materials technologist with priority-ranked quantified materials goals. In short, the output of a conceptual design study for directionally solidified composites

identifies the programs to be worked out sequentially in order to assure use in advanced engines in timely fashion. Conceptual design eliminates the need for material technologists to second guess capricious design chiefs and reduces the expenditures of materials developers on pet projects. How the need for a conceptual design approach arises can be seen by reviewing the way in which new materials, especially those which are not upgraded versions of existing ones, are committed to use.

The responsibility for committing materials for advanced engines rests with the Chief Project Engineer (CPE); materials technologists assist in an advisory capacity. Obviously, the CPE is strongly influenced by the recommendations of his materials consultants. Nevertheless, the engine and its satisfactory performance is the responsibility of the CPE and his decision to commit "new" materials is a compromise between the expected performance improvements, the cost to attain these improvements, and the risk of the "new" material failing in the engine. Realistically, of course, this decision is influenced by the customers' desires and by the need to eliminate discovered structural deficiency when no alternatives except a "new" material are available. The quantifying of the elements of this compromise is the goal of a conceptual design. It establishes for the materials technologist the ground rules for acceptability of DSC components by specifically requiring answers to the following three questions:

- How much performance improvement is to be realized?
- How much will it cost to get the performance?
- Will the material work to the predetermined life-cycle goals?

The answers to these questions or, more likely, the unavailability of answers to these questions during the conceptual design identifies specifically the programs that must be undertaken to apply the new material, in this case directionally solidified composites.

A major consideration in using any high-strength material is the usual accompanying low ductility. Considerable experience has been acquired in using relatively brittle materials in gas turbines, typically as vanes. Recognized and accepted design techniques avoid stress concentrations, utilize compression loading as much as possible, and avoid high thermal stresses. Such tricks can be attempted with DSC materials, but the anisotropic nature of the composites will cause difficulties. Imaginative design, supplemented by bench and rig testing, would be mandatory.

ASSESSMENT

Recommending a conceptual design study for DSC materials is based on a judgment that sufficient research and development have identified these materials as both useful and practical for turbine hardware. At this point in time it is clear that the "useful" identification has been made, and work is under way by several engine companies to establish practicality. The design process described in this chapter defines the materials goals and the development programs needed to reach these goals.

IX. PROCESSING OF PARTS

DSC gas turbine blades and vanes can be fabricated in the following ways:

1. Directional solidification of the item in a precision investment casting mold.
2. Directional solidification of bar stock from which parts are machined or shaped by deformation.
3. Directional solidification of airfoil shapes which are assembled into parts by joining techniques.
4. Directional solidification of sheet or wire material for subsequent assembly into complex shapes by hot-pressure bonding.

Since directional solidification is the process for developing the desired structure, the direct fabrication of the finished part in an investment casting mold is the most obvious processing route and is the approach that will be stressed below in defining processing research and development needs. To define the processing latitudes and to identify the effects of such processing on the properties of attractive directionally solidified alloys, however, exploratory work should be conducted in the areas of machining, forging, creep forming, welding, and diffusion bonding. A combination of such methods with directional solidification could prove to be the best or only way to fabricate blades and vanes from specific alloys. Similarly, the need for protective

coatings will be defined by the oxidation and corrosion resistance of specific alloys.

Directional investment casting is a production process for high-performance blades and vanes made from conventional superalloys, and a considerable amount of processing technology and equipment is directly applicable to the problem of processing composite alloys. To accelerate development and promote cost effectiveness and acceptance by engine designers, it would be advantageous to exploit this process.

A. DIRECTIONAL INVESTMENT CASTING

In defining the areas in which work is needed for the processing of DSC blades, an attempt has been made to utilize a format acknowledging the cycle through which any new material progresses in receiving qualification for engine use. It has been assumed that the alloy development effort will permit the selection of specific alloys whose properties are sufficiently attractive to warrant the significant dollar investment involved in advancing the material through bench, rig, and engine testing. Development program milestones representing processing needs can then be established and integrated with those of the engine designer to chart the course toward ultimate application.

The milestones for development of the casting process for a given DSC alloy system are:

1. Demonstration that the alloy can be directionally solidified in a complex blade shape.

2. Characterization of alloy properties in representative blade configurations.

3. Optimization of the processing method to achieve the desired morphology and properties.

4. Development of preliminary design data.

5. Determination of compositional and processing latitudes for the alloy in the particular planned application.

6. Identification of processing problems associated with transitioning from single blades to production clusters.

7. Optimization of production processing parameters.

8. Development of design data minimums.

9. Definition of production process yield characteristics as a function of imposed specifications.

10. Development of production design data.

11. Development of purchase specifications.

Bench testing of actual blade hardware can be conducted concurrently with Tasks 1, 2 and 3; rig testing would be expected to commence after Task 4 and engine testing would logically await completion of Task 8. Non-destructive testing should be initiated with the processing of the first blade and results compared with the subsequent destructive analysis of each blade during the process optimization program.

1. Demonstration of Blade Processing Capability

This task will involve a multifaceted effort. First, to determine whether the alloy can be processed in a manner analogous to the directional solidification of conventional alloy parts, mold, core, crucible, and thermal gradient and withdrawal rate compatibility should be investigated. The lack of compatibility with directional solidification capabilities that exist for conventional alloys would require the development of alternative processing procedures (i.e., new mold, core, or crucible materials and systems or different furnace designs to achieve plane front growth).

Second, variation of directional and/or equiaxed microstructure, lamellar and colony microstructure, and directional "grain" orientation control must be achieved. Demonstrating that the solidification structure can be varied is most important at this point since the desirable microstructure in terms of blade application is undefined. For example, equiaxed or transversly oriented "grains" may be desired in the root; "grains" may be desired to follow the leading and trailing edge rather than intersecting it in a purely axial orientation; and impact, foreign object damage, or thermal fatigue experiments may indicate a preference for lamellar or colony structures. Alloys that cannot be solidied to the desired structure in full-section size because of extreme gradient requirements or reactivity might be made as a sheet or wire preform and be assembled by hot-pressure bonding.

Finally, the effect of section size changes on the macrostructure and microstructure of the alloy must be identified in terms of root to airfoil, airfoil to shroud, root to thin shelf to airfoil, solid to hollow, and pin versus axial rib support in hollow designs. Problems in achieving and controlling the proper structure as a result of section size changes could justify stressing assembly procedures for part fabrication (e.g., diffusion bonding of root and shroud elements to a directionally solidified airfoil.).

2. Characterization of In Situ Properties

The *in situ* properties of DSC blades should be determined in the airfoil and across the root-airfoil junction for thick, thin, and cored configurations under processing conditions that provide lamellar, colony, and equiaxed composite morphologies. The specific properties to be measured are defined in Chapters IV and V of this report, but generally this effort should demonstrate:

a. What range of morphologies can be attained in representative blades by variation of melt temperature, thermal gradients, and solidification rate.

b. How properties vary with morphology.

c. How morphology and properties vary with part length and section size.

d. How *in situ* mechanical properties compare with the more idealized simple rod samples produced in high-purity molds.

e. How compositional effects affect property performance.

Evidence of significant compositional effects would justify the conduct of Task 5 before Task 3.

3. Optimization of Morphology and Properties

An iterative effort will be required to achieve a high fraction of the property potential of directionally solidified composites in blade form as defined by rod specimen data. This effort will also permit the tailoring of structures and properties to the requirements of various parts of the blade (i.e., airfoil, root-airfoil junction, and root). The optimization will have to concentrate on the inadequacies identified in the characterization data and should be measured against specific property objectives which could qualify parts for rig test evaluation.

4. Development of Preliminary Design Data

Utilizing optimum processing procedures, parts should be produced to yield test specimens sufficient to define the nominal shape and level of property curves considered necessary for the accomplishment of conceptional design payoff studies against specific anticipated engine mission assignments. This effort would also provide a preliminary evaluation of process reproducibility by exposing produced specimens to nondestructive and destructive analysis of structure, defects, and properties.

5. Determination of Compositional and Processing Latitudes for the Alloy

The sensitivity of the alloy to deviations from its ideal composition should be monitored in the characterization and optimization tasks. Through the preliminary design data state, high-purity materials, singly remelted and kept from exposure to excessive superheat, should be used.

Basic research to define the compositional latitudes for the primary constitutents of the alloy and to identify the maximum tolerance of the system for trace elements, which might be present in lower purity raw materials or could be picked up in the melting of the master alloy or the remelt of the casting charge, would be helpful. Effects of the trace impurities on morphological stability and on resultant properties also should be studied, and the need for special melting or remelting environments (i.e., higher vacuums or purer inert atmospheres) determined.

6. Identification of Processing Problems

Because of the need for precise control over the composite alloy structure all work to this point is expected to be conducted on single-blade elements. The automation of single element production processing, the transition to conventional directionally solidified cluster production, or the development of an entirely new production concept would be accomplished to assure that parts intended for rig and engine test would be contributing to the qualification of the process for full-scale production.

The variety of concepts for the scale-up of optimized single-blade processing parameters would be evaluated to identify problems which could be anticipated in volume production, culminating in the selection of one approach for optimization.

7. Optimization of Production Processing Parameters

Production process optimization should verify that melt temperature control, thermal gradients, and withdrawal rates can be duplicated in production and that the desired morphologies and properties can be reproduced. The process would be frozen at some optimum set of conditions, and parts would be produced for the development of design data minimums. A confirmation of process reproducibility would be accomplished and processing defect types and levels identified.

8. Development of Design Data Minimums

With a frozen production process design, data minimums would be determined to permit the design of blades appropriate for engine testing. These data should be sufficient to allow statistical definition of important design parameters and to permit conservative application of the material to a specific blade requirement.

9. Definition of Production Process Yield Characteristics

If it is to achieve acceptance for application, a blade production process must be economical. And the economics of any production process are dependent upon yield. Thus, an integral part of the development cycle

should be blade specification drafting and evaluation of the process for product yield under that draft specification.

While there are cost implications at each stage of development the matter of cost has not been emphasized in this definition of R&D needs. Nevertheless, the cost impact of decisions to be made in such a development program must receive consideration. An alloy containing large proportions of inordinately expensive elements would be difficult to justify. Incompatibilities with existing mold, core, or crucible materials would require the development of new families of refractories. And process incompatibilities with existing directional solidification equipment would require new capital investment to accomplish production of engine parts. Alloys that are intolerant of moderate impurity levels or that require narrow limits on processing conditions, such as superheat temperature, thermal gradient, or withdrawal rate, would incur greater production costs. The need for secondary fabrication processing (e.g., assembly by diffusion bonding), extensive nondestructive testing, or new and as yet undefined coating systems has distinct cost implications.

10. Development of Production Design Data

In this phase, the parameters which will be controlled during processing will be established, as will the mechanical properties for which the finished part will be inspected. Sufficient processing experience will have been gained to relate changes in manufacturing practice to the resultant quality variations of the product.

11. Development of Purchase Specifications

An important conclusion to the development of a new material or process is the development of a purchase specification that reflects the needs of the part designer and the capabilities of the part maker. The conceptual design, property characterization, process routing, and yield characteristics under the draft specification must be thoroughly analyzed in arriving at a final purchase specification for functional hardware.

B. OTHER PROCESSING PROCEDURES

Machining blades and vanes from solid rod stock is dependent upon the ability to control composite morphology in large section sizes, and upon the grinding, milling, and broaching characteristics of the alloy. Great progress has been made in electrochemical machining of complex blade configurations and work should be conducted on these alloys to determine their susceptibility to such non-traditional techniques. Machining approaches to parts fabrication would be limited to solid parts or to parts with simple cooling schemes which could be introduced by hole drilling.

Machining information is also important to achieve fit-up for the formation of parts by welding, brazing, or diffusion bonding. The solidification of a solid or hollow airfoil by directional techniques and the subsequent attachment of roots and shrounds introduce multistep processing and nondestructive testing problems that must be considered. Roots may have to be diffusion bonded to directionally solidified airfoils to sustain the axial shear loads in the

attachment. Hot isostatic pressing, casting on (by precision casting), or combinations of these may be used.

Forging or creep forming might be applicable to some alloys, especially rod morphology composites. It is likely that extreme degrees of deformation could not be tolerated, but uniform airfoil cross sections might be spread and cambered to blade shapes and roots bonded to these. Again hollow blades would be difficult to produce and the process would involve an additional major step.

Some composite alloys may have to be produced in thin sheet or wire cross section due to thermal gradient or reactivity problems. Such preform material could be assembled (much as lower temperature composites are now made) and be consolidated by hot-pressure bonding. Hollow configurations are possible but processing costs are likely to be high.

Exploratory research and development efforts in all these processing areas are justified, and comparisons might be enhanced by early standardization of machining, deforming, and joining tests so that each candidate alloy system might have a similar portfolio from which to evaluate its processing potential.

C. ASSESSMENT

While a variety of fabricating methods involving directionally solidified alloys can be used, the casting to shape in a mold is the most obvious route. The research and development cycle, typical for all new materials, has been defined.

X. BENCH AND RIG TESTING OF TURBINE BLADES

Bench and rig tests are standard procedures with new blade designs or when new materials are used in existing blade configurations. The primary reason for bench and rig testing is to learn as much as possible about new blade designs or materials in low-risk tests in order to avoid expensive surprises that can occur during full-scale engine tests.

A. BENCH TESTING

With new blade designs, bench testing often commences by testing a symmetrical model of the new design that incorporates all cooling air passages and holes. By testing a symmetrical model in the flame tunnel, thermal stresses and low-cycle fatigue can be induced so that the parts capabilities under uniform loads can be assessed. Flame tunnels can also induce mechanical and vibratory as well as thermal stresses on models, which allow for a rather rigorous evaluation.

Actual new blades, which may be new designs or new materials for standard designs, are then bench tested to determine several factors. Two examples of bench tests developed to assess blade potential as economically and effectively as possible prior to rig and engine testing are described below.

1. Natural Frequency Determination

The natural frequency of a new design or material is determined by tip excitation methods. Knowledge of the blade natural frequency is imperative in order to avoid resonant conditions which can result in blade failure. Nodal patterns and strain distributions are also determined.

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Major nodal patterns are:

1. First Flex -- bending about minimum moment of inertia,
2. First Torsion -- twisting about the stacking axis,
3. First Axial -- bending about maximum moment of inertia, and
4. Second Flex, Second Torsion, Second Axial, etc.

These patterns show how a blade vibrates and the shape it assumes under specified conditions. Strain distributions are often determined in these tests in order to determine the stress state.

2. High-Cycle Fatigue

Standard high-cycle fatigue tests are usually conducted at room and at elevated temperatures. The primary purpose of this testing is to determine how blade configuration affects material properties. This size and shape effect is extremely important since initial design data are determined from standard test specimens.

B. RIG TESTING

On completion of bench testing individual turbine parts, such as blades and other critical turbine components, are rig tested. A rig test involves a simulated engine cycle, which varies with mission requirements for the engine under consideration, for an entire turbine stage or section. During a rig test the turbine section experiences the stress and temperature environment that would occur during actual segments of a mission: takeoff, cruise, dash, and landing with thrust reversal. Emphasis is placed on the most

critical cycle segments -- takeoff, dash, and thrust reversal -- in order to maximize the severity of the test and the number of simulated missions per unit of test time.

Rig testing is considerably more complex and expensive than bench testing. However, it yields valuable component information and provides the opportunity to observe both individual parts and components under simulated service conditions. On completion of bench and rig testing, the parts undergo full-scale engine tests. Initial engine tests are heavily instrumented to determine actual engine operating conditions and to correlate with rig and bench test data.

XI. NONDESTRUCTIVE INSPECTION OF DSC MATERIALS

Nondestructive inspection (NDI) of DSC materials presents unique problems of extreme importance. No information regarding specific nondestructive tests that may be required is available, and it is highly unlikely that DSC materials will be extensively used unless nondestructive acceptance criteria that can adequately describe the structure and defects of these materials in hardware configurations are developed. On the basis of past experience with cast superalloy turbine blades, the following current applications should be readily transferable to directionally solidified composites.

	Inspection Problems	Current Inspection Method
1.	Wall thickness	Eddy current
2.	Inclusions, core remnants	Radiography
3.	Gas porosity	Radiography, ultrasonics
4.	Surface flaws	Penetrants
5.	Coating integrity	Penetrants, thermoelectric and backscatter radiation

Inspection requirements peculiar to directionally solidified composites that may be important are:

1. Grain misalignment,
2. Faults in microstructure,
3. Reinforcement imperfections or discontinuities,
4. Cellular or equiaxed structure identification; and
5. Adequacy of bond to conventional (non-DSC) structure.

Gross misalignment or absence of the reinforced composite structure in large regions of a casting may be

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determined from sonic velocity measurements by the time lapse or critical reflectivity angle methods. Also the variations in elastic moduli associated with misorientation or non-directional structure should be reflected by shifts in the frequencies of natural modes of vibration outside the band associated with the dimensional tolerances.

Local deviations of the microstructure are critical from a fracture standpoint and may require the development of new methods of detection. The anisotropy of the transport properties of these materials has not been determined but is expected to be a second-order effect. Thus the usual electrical and thermal techniques would be incapable of resolving, for example, a small region of cellular microstructure. The most promising approaches appear to be ultrasonic C-scan or acoustic holography, which would depend upon the local variation of elastic constants. Unfortunately, the composite structure and the impedance mismatch between the two phases, coupled with the random rotation of grains or colonies about the growth direction, may result in a considerable level of ultrasonic attenuation and noise. The periodic structure of a lamellar colony may give rise to a specific reflection or diffraction of sound waves at a critical frequency of excitation. Differential thermal expansion between the two phases may give rise at the free surface to a periodic topography of sufficient displacement to be detectable by optical diffraction and spatial correlation. The preferred orientation of the phases can also be measured by conventional x-ray diffraction pole figures.

Reinforcement imperfections resulting in fiber breaks or kinks under stress may be detectable from acoustic emission under mechanical stressing or from differential thermal expansion that will accompany self-induced stresses. Acoustic emission also may be enhanced by twinning of an intermetallic reinforcement or fracture of a brittle phase in the vicinity of a pore or other macroscopic flaw.

Interfacial delamination, which may occur in lamellar systems, should be resolvable by absorption of radioactive krypton. This technique has also been demonstrated to reveal subsurface porosity in conventional superalloys and should be applicable to directionally solidified composites. Thus, NDI should be made a part of any appropriate major program with directionally solidified composites, because each different system will require proper characterization of structure and properties and, in all probability, will present different inspection requirements.

ASSESSMENT

It is clear that DSC materials, like all others in an engine, will be used only if integrity can be measured and assured.

XII. NON-STRUCTURAL APPLICATIONS OF EUTECTICS

The first eutectic system to find fruitful application in devices is the InSb/NiSb eutectic as a magnetoresistive element. However, non-structural eutectics span an extremely wide range of device possibilities and the possible diversity of non-structural applications of eutectics can be seen from the set of matrix/inclusion possibilities listed in Tables IX and X. The problem is to assemble a sufficient body of basic information to enable the potential of various classes of such systems to be evaluated. This is in sharp contradistinction to the case of structural eutectics where there the goal is clear and the specific problems to be addressed are more readily definable.

A. APPLICATIONS

1. Electrical

The sine qua non of non-structural eutectic applications is the InSb/NiSb system developed by Weiss and co-workers at Siemens Research Laboratory. The major unique property of this eutectic is the high transverse magnetoresistance measured normal to the NiSb fibers. The usual magnetoresistance of InSb is effectively amplified severalfold by the secondary Hall effect of primary Hall currents carried by the conducting fibers. Commercial devices using this effect include the magnetic field probe, contactless variable resistor, current adder, and contactless push button switch. Additional applications of the same material utilize thermomagnetic effects and are discussed in a subsequent section of this report.

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TABLE IX*

Matrix of properties

The table classifies physical properties or phenomena in materials according to the kind of input parameter (colums) and output parameter (rows).

C	chemical composition, cencentration	T	temperature
E	electric field	ε	dielectric constant
H	magnetic field	χ	magnetic susceptibility
i	electric current	λ	thermal conductivity
K	(mechanical) force	μ	chemical potential
Δl	deformation	ϱ	resistivity
M	magnetic polarization	FM	ferromagnetic
n	refractive index	FE	ferroelectric
P	dielectric polarization	NE	Nernst-Ettinghausen

$\{+i\}$, etc. bracketed symbols indicate that this symbol is essential as a second input parameter (e.g., i and H in the Hall effect)

			1	2	3	4	5	6
		$\rightarrow X$	mechanical	magnetic	electrical	optical and particle radiation	thermal	chemical
	$\downarrow Y$		$K/\Delta l$	H/M	$E/P, i$	light or particle flux	T, grad T/heat current	(grad) C, (grad) μ
1	mechanical	$K/\Delta l$	elasticity (M, P, T)	1) magnetostriction 2) magnetoviscosity (suspensions)	1) electrostriction, 2) Kirkendall effect 3) electroviscosity (suspensions) 4) indirect: thermal expansion	———	thermal expansion	osmotic pressure
2	magnetic	H/M	1) piezomagnetism 2) $\mu(\Delta l)$ esp. at $T \approx T_c$	χ (Δl, T, light flux)	1) superconductors $i \approx i_c$ 2) galvanic deposition of FM layer 3) direct generation of magnetic field	photomagnetic effect	$\{+H\}$ ferromagnetic material at $T \approx T_c$	dependence of T_c on C(FM)
3	electrical	$E/P, i$	1) piezoelectricity 2) piezoresistivity	1) $\{+i\}$ magneto-resistance 2) $\{+i\}$ Hall effect 3) ac resonance effect 4) induction of voltage	ε, ϱ (Δl, M, T, light flux) $\{+H\}$ Hall effect	1) photoconductivity 2) photo-emission 3) $\{+H\}$ PEM effect 4) ionization	1) thermoelectric effects 2) $\{+E\}$ ferroelectrics at $T \approx T_c$ 3) $\{+i\}$ $\varrho(T)$	dependence of T_c on C(FE) chemical potential (grad C)
4	optical and particles	light or particle flux	stress birefringence triboluminescence	1) Faraday effect 2) magneto-optic Kerr effect 3) deflection of charged particles	1) electroluminescence 2) laser junctions 3) $n(E)$ 4) Kerr effect 5) absorption by galvanic deposits 6) cold emission of electrons	n (Δl, M, P, T, E) fluorescence scintillation colour-centre activation	thermoluminescence ———	chemoluminescence ———
5	thermal	T, grad T heat current	1) heat of transition of pressure-induced phase transition 2) piezoresistivity + Joule heating	1) adiabatic demagnetization 2) $\{+i\}$ grad T NE effect 3) $\{+E\}$ magneto-resistance effect + Joule heating	1) dissipation in resistance 2) Peltier effect 3) $\{+H\}$ grad T NE effect	absorption	λ	reaction heat
6	chemical (grad) μ	(grad) C, (grad) μ	pressure-induced phase transition	———	1) electromigration 2) galvanic deposition	light- or particle-stimulated reactions (photosensitive layers)	1) Soret effect (grad T) 2) phase transition (T) 3) change of chemical equilibrium (T)	———

* Albers, W., 1973; Van Suchtelen, J., 1972.

TABLE X*

Product properties of composite materials

X–Y–Z (Table IX)	property phase I *X–Y*	property phase II *Y–Z*	result *X–Z*
123	piezomagnetism	magnetoresistance	piezoresistance; phonon drag
124	piezomagnetism	Faraday effect	rotation of polarization by mechanical deformation
134	piezoelectricity	electroluminescence	piezoluminescence
134	piezoelectricity	Kerr effect	rotation of polarization by mechanical deformation
213	magnetostriction	piezoelectricity	magneto-electric effect
213	magnetostriction	piezoresistance	magnetoresistance: spin–wave interaction
253	Nernst–Ettingshausen effect	Seebeck effect	quasi-Hall effect
214	magnetostriction	stress-induced birefringence	magnetically induced birefringence
312	electrostriction	piezomagnetism	electromagnetic effect
313	electrostriction	piezoresistivity	coupling between ϱ and E (negative diff. resistance, quasi-Gunn effect)
343	electroluminescence	photoconductivity	
314	electrostriction	stress-induced birefringence	electrically induced birefringence light modulation
421	photomagnetic effect	magnetostriction	photostriction
431	photoconductivity	electrostriction	
434	photoconductivity	electroluminescence	wavelength changer (IR–visible, etc.)
443	scintillation	photoconductivity	radiation-induced conductivity (detectors)
444	scintillation, fluorescence	fluorescence	radiation detectors, 2-stage fluorescence

* Albers, W., 1973; Van Suchtelen, J., 1972.

Workers at Philips Laboratories have made semiconducting eutectics, like $SnSe/SnSe_2$, in which each interface is a p-n junction. Specific devices utilizing these heterojunctions have not been announced as yet.

The possibility of producing superconducting materials with improved properties by the directional solidification of eutectics was investigated quite early, but no unambiguous enhancements in critical fields or currents have been found. Indeed, in metal-metal systems involving one superconducting and one normal phase, a net decrease in the critical temperature and critical field of the superconducting phase is to be expected. Similarly, in the case of Type I superconductors, the positive surface energies of superconducting-normal boundaries will result in a decrease in the critical field of fine specimens with non-zero demagnetizing factors. If a superconducting phase is in contact with a nonconducting phase, however, then decreasing particle size leads to an increase in critical field, but this effect has not yet been observed in a eutectic alloy.

Other electrical applications of aligned eutectics are still in the speculative stage of development. Electrical conductors for critical applications might be strengthened by eutectic growth. For example, an aligned Al/Al_3Ni eutectic has been prepared to circumvent the detrimental piezoresistance of pure Al at low temperatures. A lamellar eutectic of two metallic phases separated by a high dielectric phase might have advantages as a capacitor. Furthermore, the unidirectional conductivity of insulator-metal eutectics may have some applications, such as for substrates with grown-in electrical contacts.

2. Magnetic

Sufficiently thin fibers of a soft magnetic material will be single domains with high shape anisotropy. Attempts to make permanent magnets from aligned eutectics have not been outstanding because the fiber diameters have been too large. For example, a coercive force of 10 Oe was obtained with 1.5μm Fe rods in an FeS/Fe eutectic and coercive forces of 12 and 18 Oe were achieved with 1.5 and 1.0μm rods in Fe_xSb/Fe eutectics. Using a high-growth-rate technique Livingston achieved a coercive force of 330 Oe in an Au/Co eutectic. Subsequent wire drawing increased the coercive force to 925 Oe. His analysis shows that growth rates of order 1 cm/sec will be necessary to approach the ultimate coercive force attainable by shape anisotropy.

The need for ultra-thin fibers for high coercivity is relaxed somewhat if the fiber phase has some crystal anisotropy. Here it is necessary to grow the fibers parallel to their easy magnetic axis. The few existing experiments involving eutectic phases with crystal anisotropy have not been successful in some cases because of the orientation requirement and in others because the volume fraction of magnetic phase was too small. Conceivably a high-energy product magnet could be obtained by eutectic combination of hard and soft magnetic materials. One might also use exchange anistropy between ferromagnetic and ferrimagnetic phases to achieve a useful magnetic eutectic, but no experiments of this sort have been reported.

Soft magnetic materials may be mechanically strengthened by eutectic fibers for high-speed, high-temperature rotors.

Experiments on iron modified cobalt base eutectics have achieved the desirable strengthening at the expense of introducing mechanical and magnetic anisotropy.

A summary table of eutectic systems which have been investigated is available. To date, however, no system has been found which offers significantly improved permanent magnets. One favorable area presently appears to be the development of soft magnetic materials with improved high-temperature strengths. Both areas have by no means been extensively evaluated, however.

3. Thermal

Directionally solidified eutectics have anistropic thermal conductivities substantially different from that of the matrix phase. For instance, the thermal conductivity of a UO_2/W directional eutectic, parallel to the tungsten fibers, is about 30 percent greater than that of pure UO_2. InSb/Sb directional eutectics, on the other hand, have lower thermal conductivity than either pure phase. In the latter case the anisotropy in conductivity is attributed to phonon scattering from the fiber boundaries. In some cases the Peltier effect of spontaneous thermoelectric currents between the phases may effectively enhance the heat transport parallel to the eutectic axis.

The thermoelectric power of directional eutectics is also anisotropic. Data have been reported for InSb/Sb, Te/Bi_2Te_3, Mg/$Mg_{17}Al_{12}$ and Bi/Cd composites. In each of these cases the figure of merit for thermoelectric applications is less than that of the pure matrix phase so that thermoelectric applications are not promising.

The InSb/NiSb directional eutectic has substantial thermomagnetic effects for temperature gradients parallel to the fiber axis, but its thermal conductivity is too high to permit practical use as an energy converter. However, the thermomagnetic effect contributes to the operation of a useful infrared detector described in the next section.

4. Optical

Unique optical effects occur for conducting fibers grown in a transparent matrix. When the fiber diameter is small and the length is at least comparable to the wavelength of the radiation, the composite can serve as a polarizing filter for radiation travelling perpendicular to the fibers. Siemens produces such an infrared polarizer from the InSb/NiSb directional eutectic. A eutectic with regularly spaced conducting fibers could be used as a two-dimensional diffraction grating, but apparently has not yet been achieved. Another, as yet unrealized, application is for an electron beam to optical transducer.

The InSb/NiSb eutectic has also been developed by Siemens into a broadband infrared detector operable at room temperature. Radiation directed parallel to the fibers is rapidly absorbed and the resulting thermal gradient and an orthogonal magnetic field generate the electrical output. The NiSb fibers evidently increase both the spectral response and detection efficiency of the InSb matrix. The same eutectic system may have use as a light source based on the recombination radiation of injected carriers.

Fiber optic properties are possible with directionally solidified eutectics of transparent phases with dissimilar

refractive indices. Some alkali halide eutectics have been grown to demonstrate this effect.

5. Other Possible Applications

In addition to the broad general categories described above, there exist also several more specific categories of uses that appear promising. Contrasting chemical properties of eutectic phases have been utilized in some applications. Selective etching of the fiber phase of a directional eutectic can produce a filter with a high density of micron size pores. This has already been done successfully in the case of NiAl/Cr and NiAl/Mo eutectics. Uniform holes, 0.5 microns in diameter, were produced with densities as high as 8.5×10^7 holes/cm^2. Conversely, etching the matrix phase of a fibrous eutectic produces a surface with protruding needles. In one study, the field emission from such a surface prepared from a Ni/W eutectic broke down at a relatively low current density (2×10^{-4} A/cm^2) because the fiber spacing was not sufficiently uniform. Field emission current densities of 10^{-1} A/cm^2 have also been obtained using UO_2/W eutectic structures. This latter system offers, in addition, the possibility of providing fuel elements with an anisotropic thermal conductivity, as discussed above. Suggestions have been made for other possible applications utilizing chemical properties. For instance, one might use corrosion resistant lamellae to protect another phase from corrosion. Certain eutectics conceivably may have surface states useful for catalysis.

Elastic, acoustic, and damping properties of directional eutectics are expected to be both anisotropic and unlike the same properties of the constituent phases. Eutectics

of semiconducting and piezoelectric phases may make useful acoustic waveguides, transducers or amplifiers. The possible control of thermal expansion by an aligned eutectic structure has been suggested. However, actual application of any of these possibilities has yet to be demonstrated.

B. ASSESSMENT

It is clear that most of the present applications utilize a response of one of the phases to a single generalized thermodynamic force (electric field, magnetic field, stress, temperature gradient, etc.). The important exception to this statement, namely the InSb/NiSb eutectic developed by Weiss and co-workers, utilizes the coupled response of both phases to at least two general forces. The difficulty of conceiving other easily grown, useful combinations of comparable utility is attested to by the continued sole eminence of the InSb/NiSb devices as practical non-structural eutectic materials over the nine years since the first of this family of devices was reported. Nevertheless, the potential of eutectic materials for non-structural applications is so large and diverse that the existence of other systems with unique property capabilities can confidently be predicted. A matrix of possible applications is shown in Table IX. The very scope of the field may be at one and the same time its greatest advantage and its greatest drawback. What is needed most, perhaps, is a continuing systematic and experimental evaluation of potential systems and applications carried out by interdisciplinary groups, possessing adequate depth in both materials science and device engineering capability.

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Potential applications of a non-structural nature are covered in a series of publications (Bever, et al.; Galasso, 1957, 1970; Van Suchtelen, 1972).

XIII. CONCLUSIONS AND RECOMMENDATIONS

1. Directionally solidified composites are of particular merit because they can be used at high-strength levels at a very high fraction of the melting point of the alloy system. The current estimate of the increase in temperature capability of DSC materials compared to that of the best conventional nickel- and cobalt-base superalloys is at least 100 °F (50 °C). While there are materials which already substantiate this observation, there is no one alloy that encompasses all the desired and necessary properties to qualify the material for gas-turbine-blade applications.

2. Directionally solidified composites are a new class of materials possessing unusual and highly anisotropic microstructures and properties; they have already found use as magnetoresistive and infrared materials. Broad support of fundamental research in directionally solidified composites is recommended, including non-structural considerations, in view of the wide variety of applications that these materials are potentially capable of addressing.

3. Conceptual design studies are recommended for specific DSC materials and specific engine applications to establish quantitatively the pay-offs possible and the cost and performance criteria necessary to justify their use.

4. There is great need for a significant increase in alloy development studies; this stands out as perhaps

the major conclusion. While several of the alloy systems of current interest show great promise for high-temperature applications, it appears that oxidation and corrosion within these systems will remain a continuing problem. Alloy systems utilizing a ductile matrix, or alloys in which one of the phases is ductile, deserve major support, especially in view of their probable greater resistance to thermal and mechanical shock and because of their greater similarity to conventional current alloys in anticipated applications. The potential for oxidation resistance is greater in those alloys where the ductile matrix phase can be suitably alloyed. Typical of the needed studies are the following items:

a. Phase diagram studies should be an important part of such alloy development programs.

b. Latitude of compositional tolerances should be examined in order to be able to change the volume fraction of the phases (off-eutectic compositions).

c. The role of impurities must receive adequate attention in each alloy system studies (see item 5).

d. Research should include systems utilizing oxides, carbides, nitrides, silicides, and borides, because of their indicated greater resistance to erosion and anticipated resistance to either oxidation, corrosion, or both. In these systems, in particular, useful phase diagrams are lacking.

e. Some attention should be given to higher melting point systems; this will require intergrated studies of melting capability, mold and core reaction problems, and process control. Because of anticipated casting problems, alternate fabrication and processing routes may be necessary.

5. Emphasis should be placed on impurity effects as they influence the casting process as well as the stability of structure and properties. Some evidence exists that the effects of impurities on stability may be large, and that the effect becomes larger for the higher cooling rates which produce the finer, stronger structures.

6. For alloys of current interest among directionally solidified composites, it appears that precision casting techniques with modifications, may be applicable. All of the lower melting point systems will not be inert to the ceramic materials utilized: indeed, for the slow solidification rates, reactions with both the precision casting mold and the cores may be an important problem. If systems of somewhat higher melting point are found to be potentially useful, casting problems will probably be encountered that will require new improved casting techniques.

7. It is imperative that at an early stage of DSC development, several of the better alloys be processed extensively by precision casting techniques, both in simple shapes (round and triangular or diamond cross sections) and in prototype blade shapes at a later

date, to establish property and structure levels under a variety of conditions. Taper and twist, section size, section size change, and other geometric factors must be studied. Reproducibility of results must be established both in terms of structure and properties. Inspection techniques should be sought, developed, and applied at all stages of the program, with extensive sectioning of rods and blades to provide test bars from various locations for structure and property determinations.

8. Characterization data qualify particular alloys for further study and development. The overall test program should include all of the mechanical and physical tests that will be demanded by designers to advance the materials for bench, rig, and engine tests.

   Even before specific alloy systems are selected for the more extensive development and parts testing, several typical DSC alloys should be studied in detail to establish modes of deformation and fracture over a range of stress and temperature conditions. Required as soon as possible are creep rupture data to classify values such as instantaneous plastic strain on loading, first stage transitional creep, magnitude and rate of second stage creep, elongation at the end of second stage creep, and total ductility. From these data one can provide designers with the values of the time to achieve 0.1, 0.2, 0.5, and 1 percent plastic strain over a range of temperatures

should be initiated to permit study of the mechanisms of deformation and fracture. Impact and fracture toughness data should be generated to qualify any alloy for further study. Off-axis properties should receive emphasis. In addition to measuring the aforementioned structural properties, it is necessary to determine the altering of these properties in service environments. For example, foreign object damage causing localized plastic deformation could result in a recrystallized structure exhibiting sharply diminished high-temperature strength.

9. Long-time stability studies, preferably with coated materials in simulated environments, should be undertaken to determine the long-time, high-temperature breakdown of the structures. Thermal cycling and thermal gradients should be examined for their effects.

10. Oxidation- and corrosion-resistant coatings should be examined early in the development effort. Some preliminary work appears reasonably promising and certainly encourages further work. In particular, studies should examine the mechanism of oxidation and corrosion to establish whether damage occurs frontally, along interfaces between phases, or down specific phases. The role of phase dimensions and the relative volume fractions of the phases should also be examined for their effects on oxidation and corrosion rates.

11. Programs should be undertaken to anticipate the joining problems of the several classes of candidate materials. In particular, these studies should consider the problems of blade attachment to discs. Since the blade root will probably not be a fir-tree structure directly impressed on the DSC material, techniques must be sought to solve the attachment problem (e.g., the casting on of a root section, the hot-isostatic pressing of a root attachment, the diffusion bonding of a preformed root, or combinations of these.). The properties of such joints must be determined for blade loading conditions.

12. Machining, grinding, and other material removal methods for application to DSC materials are recommended for study.

13. Nondestructive testing should not be considered initially as a separate R&D feature but should be made part of the earliest alloy development and processing programs. Because of the many unknowns regarding faults and defects in the various classes of alloys, nondestructive evaluation techniques should be considered at the onset of any program. The area of NDT is perhaps the area of greatest unknowns pending the establishment of a performance base.

14. DSC materials today are in an early stage of alloy and processing development. Although progress has been good and technological promise is real, development is not yet sufficient to warrant a multi-million dollar engine program limited to any particular alloy or class of alloys.

In addition to the developmental programs advocated above, a continuing basic research effort in DSC materials should be pursued to insure a proper base for the development of practical materials.

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