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CANADA



DEPARTMENT OF MINES AND TECHNICAL SURVEYS

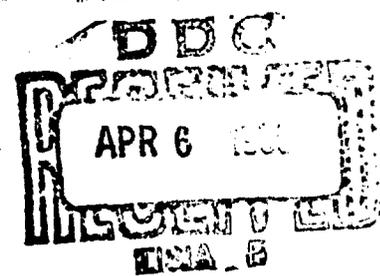
MINES BRANCH

OTTAWA

PHYSICAL METALLURGY DIVISION

RESEARCH AND SPECIAL PROJECTS REPORT FOR 1965

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## INTRODUCTION

During the past year, the Physical Metallurg. Division has continued to pursue various lines of research and development pertinent to the sustained growth of Canada's metallurgical industries.

Some of these projects have been initiated by the armed services, in order that they might develop weapon systems best suited to Canadian requirements and environment. Others have been selected from the point of view of assisting the metallurgical industries in their continuing improvement of production and fabrication facilities for metals such as iron and steel, aluminum, copper, magnesium, etc.

Yet another type of research and development is intended to provide long term technical guidance to the growth of the industrial effort into those areas of processing and fabrication associated with the newer metals and alloys of both the ferrous and non-ferrous types.

No research establishment of this nature is well balanced unless attention is also paid to research of a fundamental nature. Accordingly, an appropriate portion of the effort is directed towards those subjects that are basic to a clearer understanding of the scientific phenomena underlying projects of an applied nature that form the bulk of our activities.

S. L. Gertsman  
Chief of Division

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## ALLOY RESEARCH

Alloy research in this Division comprises a broad exploratory and development field of endeavour directed toward the improvement of available materials as well as the search for new uses and applications for products based on the abundant natural resource metals produced by the Canadian mining and metallurgical industry.

### Effects of Hafnium on Carbon Structural Steel

In the continuing study of minor additions to carbon steels, attention has been focused on elements of the IV-B and V-B group. The effect of the first four members (titanium, zirconium, vanadium and niobium) are fairly well known and, in spite of their general chemical similarity, each is significantly different in its influence on carbon steel. The influence of hafnium, another element of the IV-B group, on the properties and structures of 0.14% carbon, 1.20% manganese, structural steel is currently being studied. Preliminary results have been obtained on six 50 lb induction melts comprising two hafnium-free ingots as reference standards, and four ingots containing 0.04%, 0.08%, 0.15% and 0.20% hafnium, respectively. In the as-rolled state the two lower levels of hafnium had no effect on the 15 ft-lb Charpy V-notch transition temperature nor on the yield point. The two higher levels raised the transition temperature slightly and the yield point was eliminated.

There was no significant variation in ultimate tensile strength. Tensile ductility increased slightly with increasing hafnium contents. Normalizing changed the effects to a significant degree in only one respect: all the steels showed a yield point, whereas the yield point was eliminated at the two higher levels of hafnium in the as-rolled condition.

Quantitative metallographic study shows that hafnium alters the microstructure slightly. In the as-rolled condition, there is an irregular trend towards a decreasing percentage of pearlite with increasing hafnium contents. This trend is not shown after normalizing. In common with many elements, hafnium has the effect of disrupting the formation of lamellar pearlite, causing the formation of what is usually termed divorced pearlite. With increasing hafnium contents, not only is the carbide within the patches of divorced pearlite more discrete, but there is a greater percentage of isolated single carbides. Tentatively, it is considered that the measured drop in percentage pearlite is ascribable to failure to detect and measure these isolated, fine, cementite particles rather than to the formation of hafnium carbide.

With respect to grain size in the as-rolled state, 0.04% and 0.03% hafnium refined the grain size somewhat, but at the 0.15% and 0.20% levels the mean linear intercept grain size returned to the level of the hafnium-free reference melts. After normalizing, 0.04% hafnium reduced the grain size very slightly, but higher contents coarsened it significantly.

A preliminary assessment of the form of the non-metallics showed that 0.15% and 0.20% hafnium eliminated the stringer type inclusions, whereas no such effect was brought about by lower hafnium levels. At the two higher hafnium contents, numerous cubic inclusions ranging in colour from lemon yellow to reddish yellow were observed. These are markedly similar to those formed by zirconium.

#### Molybdenum and Temper Embrittlement

The general effects of molybdenum on the ductile-brittle transition temperature and temper embrittlement of quenched and tempered steel have been well documented. The results of experimental work done on AISI 3135 steel and reported last year were, in some respects, at variance with the published literature.

Testing of a second series of steels, similar to the steel noted above, but with a slightly higher nickel content, confirmed the earlier results. Another series of larger ingots of 1.8% Ni, 0.6% Cr steels with six molybdenum levels ranging from residual to 1.0% have been prepared. For this series, considerably more material will be available, thus allowing a more complete determination of properties and a more quantitative evaluation.

The Effects of Small Additions of Titanium  
and Zirconium on the Tempering of a Low Carbon 3% Cr Steel

Two steels, with and without titanium and zirconium, are being used to study the effects of these elements on the tempering characteristics of the steel.

In the normalized condition the steels are bainitic with about 30% primary ferrite. The carbide in the bainite is very sparsely distributed and is dissolved by most etching reagents.

On tempering, the steel without titanium and zirconium shows a secondary hardening reaction at about 565°C (1050°F) with the formation fine of fine needles of a carbide of  $M_{23}C_6$  type. The needles grow preferentially in the bainitic areas where there is more carbon than in the primary ferrite.

In the steel with titanium and zirconium relatively few needles are seen after tempering at 565°C (1050°F). The tempering seems to be retarded so that it is necessary to temper 10-40°C (18-72°F) above the temperature used for the steels without titanium and zirconium to obtain comparable structures. However, there are always fewer needles in the steel containing titanium and zirconium.

The distribution of carbides after tempering is very sensitive to the thermal and mechanical treatment before tempering, and can be changed radically by rolling or forging. Further, the differences between the structure of the two steels change with pre-tempering treatment.

A systematic analysis is therefore being undertaken, starting with quenched and tempered structures.

The presence of titanium and zirconium also decreases the impact resistance of the steel but no direct cause for this has been observed.

#### Uranium in Steel

The effect of a range of uranium contents on the morphology of sulphide inclusions in Type 416 and Type 430F stainless steel containing 0.15% and 0.30% sulphur and three levels of uranium contents, to give uranium/sulphur weight ratios of 1:1, 4:1, and 6:1, was examined in relation to such properties as transverse ductility and cold formability by cold upsetting, heading, and cold swaging. It was found that a uranium/sulphur ratio of 4:1 brought about a marked improvement in all these characteristics.

During the past year the influence of the uranium/sulphur ratio on corrosion and forgeability of these steels was investigated.

Corrosion testing has shown that the addition of sufficient uranium increased the corrosion resistance of these steels in nitric acid to a level very similar to the equivalent non-resulphurized steel. It was postulated that this demonstrates a change in the chemical composition of the sulphides.

Forgeability tests were not conclusive. In the industrial production of large ingots of resulphurized steel, manganese does not completely prevent the formation of low-melting point sulphides, thus forging difficulties are experienced. In a special series of simple carbon steel heats in which the sulphides of iron, manganese and uranium were produced, the greater sulphide-forming ability of uranium over iron and manganese was demonstrated. It was also demonstrated by hot-torsion forgeability tests that improved forgeability was associated with this sulphide. However, similar tests on as-cast test bars of uranium-bearing resulphurized bars of AISI type 400 stainless steels have been inconclusive. The testing technique used is not sensitive enough to measure any differences in forgeability that might exist.

#### Aluminum Casting Alloys

In connection with earlier work on premium quality castings, a review of high-strength aluminum casting alloys revealed the possibility of considerable improvement of their mechanical properties. An exploratory investigation of high-purity alloys based on Al-4% Cu resulted in unusually high tensile properties (up to 72 kpsi UTS, 70 kpsi 0.2% YS, 5% El). Further work on this and some other aluminum alloy systems is being continued.

### Magnesium Alloys

Research on Mg-Zn-Ag-Zr casting alloys was continued to determine the minimum silver content required in ZQ71 and ZQ91 alloys consistent with high properties. Preliminary investigation revealed that high properties are obtainable with as low as 0.2% Ag in ZQ91 and 0.4% Ag in ZQ71 alloys.

Present work is connected with an investigation of the effect of small amounts of deformation by press-forging on the mechanical properties of castings and a study of forgeability of some high-strength magnesium alloys.

### Copper Alloys

#### 1) Aluminum Bronzes of High Strength and Ductility

Complex nickel-aluminum bronze casting alloys have been examined in detail to determine the solubility and composition of the secondary phases controlling the mechanical properties of the material, in particular yield strength and elongation. Metallographic examination and chemical extraction methods showed that the two important compounds  $Fe_3Al$  and  $NiAl$  are completely soluble in the high temperature beta phase up to 5% Fe at 1000°C and up to 7% Ni at 950°C.

An interaction exists such that the phase field alpha + beta + Fe<sub>3</sub>Al is reduced by increase in nickel content, while the field alpha + NiAl is unaffected by iron content. The precipitation reactions occurring during cooling of cast aluminum bronze are therefore dependent on both the iron and the nickel contents. At high iron content, Fe<sub>3</sub>Al separates continuously from the beta phase down to 600°C. The residual iron in the alpha phase at this temperature is 1 wt %. NiAl appears as a result of the eutectoidal decomposition of the nickel-rich beta phase. Thermal analysis showed a eutectoid reaction temperature of 700°C at 2% Ni and 910°C at 5% Ni.

X-ray examination of chemically extracted residues showed that structurally, Fe<sub>3</sub>Al and NiAl were almost identical, with b.c.c. lattice spacings of 2.895Å and 2.904Å respectively. (It was also shown that the NiAl existed as a super-lattice in the slow-cooled condition, while no ordering was evident in Fe<sub>3</sub>Al).

The eutectoid reaction yielding NiAl has been shown to affect adversely the tensile ductility of cast alloys. Additions of 1% antimony or tin to high nickel-aluminum bronzes tended to alleviate this problem by producing a granular rather than a lamellar NiAl eutectoid phase. The significance of this structural modification is being investigated in an attempt to evolve high nickel cast bronzes with yield strengths of 45-50 kpsi and about 20% elongation to ensure sufficient ductility.

2) Aluminum Bronzes of Improved Strength by Various Hardening Mechanisms

Binary alpha phase copper-aluminum alloys also provide a possible basis for the development of materials susceptible to age-hardening, work-hardening, and structural hardening by transformation mechanisms. Exploratory ageing experiments have been conducted on 7%, 10% and 12% aluminum bronzes each containing one of the following additions:- 0.5% Zr, 0.5% Be, 0.5% Cr, 10% Ni, and 1% Sn. The hardness testing of heat treated samples taken from rolled plate has indicated the possibility of a precipitation hardening mechanism existing in alpha Cu-Al alloys containing zirconium, beryllium, and chromium. The modified alpha/beta and beta aluminum bronzes are more complex in that the hardening associated with martensitic decomposition tends to obscure the effects of ternary additions. Using tempering times of one hour, a hardness peak at 240 DPH was detected at 500°C for the 12% Al binary alloy. The effect of alloying elements on this peak and on the beta-martensite transformation is being investigated.

Effect of Uranium Additions to Copper Alloys

1) Wear Resistant Electrical Conductors

Earlier work had indicated that small quantities of uranium added to copper gave increased hardness and improved wear resistance with little deleterious effect on the electrical conductivity. It was considered therefore that this might have applications in the electrical field for conductors where sliding friction was involved.

An apparatus was therefore constructed to test the abrasion wear resistance of dilute copper-uranium alloys. Preliminary experiments established those parameters, in particular humidity, tending to affect reproducibility and a procedure was established for abrading 3/16 in. diameter wire on 600 silicon carbide paper moving at radial speeds of 2000 and 5000 inches per minute. Runs of one hour duration at the faster speed gave weight losses of 0.0310 grams for electrolytic tough-pitch copper, 0.0275 grams for a 0.11% U copper, and 0.0255 grams for a 0.25% U copper. Tests will be continued on 0.5% and 1.2% U coppers.

## 2) Heat Treatable Alloys

The use of uranium as a deoxidant and alloying agent in zirconium coppers is being investigated in induction air melted 50 lb heats. Compositions of nominally 0.20% Zr with and without uranium deoxidation and with and without an addition of 0.5% U have been chill cast into slab moulds and rolled to  $\frac{1}{2}$  in. plate. The results to date have shown a 65% recovery of zirconium without uranium deoxidation and 90% recovery with prior uranium deoxidation. These materials have been processed for heat treatment and property evaluation.

## Alloy Metallurgy of the Refractory Metals

In the investigation of possible dispersion-hardening in niobium-hafnium-carbon alloys the range of composition under study has been extended to include up to about 6 at.% Hf and 4 at.% C. By metallography and X-ray diffraction two

carbides have been identified in the system,  $Nb_2C$ , and the monocarbide  $(Nb, Hf)C$ , which is isomorphous with  $NbC$  and  $HfC$ . With increasing hafnium content of the alloys  $(Nb, Hf)C$  replaces  $Nb_2C$  as the stable carbide.  $Nb_2C$  forms as massive particles and contributes only minor hardening. The monocarbide forms as fine, uniformly dispersed particles at  $1200-1400^\circ C$  ( $2190-2550^\circ F$ ), but is subject to solution and probably agglomeration with increase in temperature; thus, the thermal stability is lacking that would be necessary to effect useful dispersion-hardening at high temperatures. However, a mild ageing response is associated with precipitation of the monocarbide at  $1200^\circ C$  ( $2190^\circ F$ ) in some alloys.

Certain compositions when cooled rapidly from high temperatures yielded a phase that could not be identified as any known compound. Work is continuing in order to identify this phase, its structure and its occurrence. A computer programme has been located to assist in the determination of the crystal structure from X-ray diffraction data. To make possible faster specimen cooling rates, a modification has been made to the high-temperature vacuum annealing furnace.

#### Constitution of the Titanium-Aluminum System

A critical survey of the literature concerning the constitution of the titanium-aluminum binary system has shown the existence of much contradictory evidence about the form of the diagram. In the area between 16 wt % and 23 wt % Al there is considerable controversy over the possible existence of an epsilon phase.

Recent Mines Branch work on the titanium-aluminum-molybdenum system indirectly supports the case for the existence of this phase.

Because accurate knowledge of the titanium-aluminum binary diagram is essential to the understanding of all higher order titanium alloy systems involving aluminum, a study of this binary system has been commenced, starting with the area between 15 wt % and 25 wt % Al.

Six titanium alloys within the above range have been prepared for long-time annealing and special annealing facilities have been designed and installed.

#### Uranium-Base Alloys

Work was completed on the binary alloy portion of a uranium alloy development programme on high density alloys.

A standard procedure was established to produce cast test rods and the following uranium-base alloys were prepared, heat treated and tested: 1, 2 and 3% Mo, 1, 2 and 3% Nb, and 3% V, 0.5 and 2% Zr, and 0.5 and 2% Ti. Tensile and compressive strengths of the alloys in the as-cast condition gave values ranging from 128 kpsi (U-2% V) to 195 kpsi (U-3% Nb) for the ultimate tensile strength, and 78 kpsi (U-1% Mo) to 161 kpsi (U-3% Nb) for the 0.2% compressive yield strength. Only the U-3% Nb alloy exhibited a definite ultimate compressive strength, which was 396 kpsi.

Hardnesses ranged from 260 VHN (as-cast) to 680 VHN (heat treated). Although some of the alloys can be heat treated to give over 500 VHN, their low ductility limits their usefulness and is thought to be due to a hard, brittle phase formed during ageing.

Tests carried out on low carbon (200 ppm) and high carbon (1600 ppm) U-2% Mo alloy indicated that, although the high carbon content may reduce the as-cast ductility, when the alloy is heat treated to maximum hardness it exhibits low ductility irrespective of the carbon content within this range.

#### Zirconium Alloys

Further investigation was made into the reason for changes in corrosion behaviour of zirconium-copper-molybdenum alloys with varying heat treatments. Results of previous attempts to identify an intermetallic compound that precipitated during ageing in a Zr-1.0 Cu-1.5 Mo alloy were inconclusive because there was insufficient present. However, X-ray diffraction analysis of a Zr-3% Cu-4.5% Mo alloy after quenching from 995°C (1820°F) and ageing at 500°C (930°F) for 24 hr and 100 hr showed, in addition to the alpha zirconium, a cubic phase that remained unchanged with ageing time. This phase is not recorded in the literature.

To establish a more complete identification, specimens containing this phase are being examined by an electron probe analyzer. Preliminary results show the intermetallic to be a ternary phase and to consist of 15% Cu, 30% Mo and 55% Zr.

## MELTING AND REFINING

### Vacuum Degassing and Casting of Steel

Hydrogen and nitrogen contents have been determined for fifteen acid electric steel melts at the C 70-1.20% Si level and for a similar series at the 0.30-0.70% Si level in the non-degassed and ladle-to-ladle degassed conditions. The metal was cast in green sand moulds and the castings were sectioned and etched to determine the proportion of equiaxed sections. The gas level throughout melting and after degassing was also determined for steel melted by single slag basic electric practice.

A series of vacuum-deoxidized high-strength nickel-cobalt steel melts, having carbon contents of 0.05%, 0.25% and 0.45% and silicon contents of 0.05%, 0.20% and 0.40%, have been cast in 1 in. and 6 in. sections for evaluation of mechanical properties. Vacuum-deoxidation has facilitated casting of sound sections at low silicon contents and use of this technique is being extended for comparison of AISI 4340 steel having 0.1% to 0.05% Si with air melted AISI 4340 steel.

Three additional vacuum-stream-degassed ingots of low-silicon, Cr-Mo-V steel are being cast to supplement a previous series of vacuum-cast steel ingots.

### Effect of Pouring Temperature and Holding Time on Properties of Sand-Cast Magnesium Alloys

The influence of pouring temperature and holding of the melt before pouring on the mechanical properties and chemical

composition of several magnesium casting alloys has been investigated. The results show that variations in pouring temperature from 700 to 850°C (1290 to 1560°F) and in holding time for up to two hours in the molten state did not adversely affect the properties to a pronounced degree.

The "insoluble" zirconium content may be appreciably decreased by lowering the melt temperature to 700°C (1290°F). Alloy ZH62 should not be heated to 850°C (1560°F) because of its drossing tendency. The "soluble" zirconium and rare earth contents of alloy QE22, the rare earth content of alloy EZ33 and the thorium content of alloy ZH62 may be decreased by holding the melt at normal pouring temperature for several hours.

## CASTING

### Solidification of Steel

Apparatus for controlling variables associated with the solidification of laboratory melts of Ferrovac E has been modified to use a back-pressure of argon to avoid vaporization losses and difficulties. It has been found possible to continuously measure temperatures at several positions within the melts and so to measure growth rates and thermal gradients. Techniques of adding  $P^{32}$  and handling the radioactive melts and ingots are being developed.

### Efficiency Tests on Differently Shaped Risers for Steel Castings

Theoretically, the ideal shape of a riser is a sphere, since it is this shape which has a maximum volume for a given surface area. This has not been a practical shape for a foundryman to utilize until the advent of expanded polystyrene. This material can be moulded in the sand and will burn out on contact with molten metal. However, because of the small area of contact of a sphere with the casting, premature freezing of the neck would result. A knock-off core has been designed in which a small amount of exothermic material is placed in the neck area and which acts to keep the neck molten sufficiently long to allow the risers to feed with no premature solidification. Castings are currently being produced which are risered either conventionally or with polystyrene spheres, pear shapes, or exothermic

sleeves around a cylindrical riser. Soundness of the 4-in. section castings is being checked by gamma radiography of the 4-in. section, ultrasonic inspection of the 4-in. section and radiography of a  $\frac{1}{2}$  in. thick slice removed from the centre line of the 4-in. section. In this way, a correlation between the three techniques and the relative sensitivities can be checked, as well as the relative efficiencies of the risers in question.

#### Water Spray Chilling of Aluminum Alloy Shell Mould Castings

An investigation of the properties of various aluminum alloys cast in shell moulds showed that alloys with long freezing ranges tend to exhibit low properties when cast in these moulds. This is due to the weak chilling effect caused by the low thermal conductivity of the mould material, which results in low thermal gradients, and non-directional solidification.

A novel method of chilling shell mould castings by the external application of water sprays has been developed and investigated using C4, SC51, SG 70 and G10 aluminum alloys cast into plate and test bar moulds. These alloys were chosen so as to give different freezing ranges since it is known that cooling conditions are more critical in the case of alloys with long freezing ranges (G10).

The tests have shown that the spray chilling method overcomes the adverse effects of the thermal characteristics of the shell mould material and gives results that are comparable with those obtained by conventional chilling in green sand moulds.

As anticipated, this improvement in properties brought about by spray chilling was most pronounced in the alloys with the longest freezing range. Thus, unchilled shell cast G10 had very poor properties, whereas the same alloy spray chilled had properties at least as good as the chilled green sand castings. In the case of SG70 (short freezing range) spray chilling again produced improved properties, but here the range in properties between chilled and unchilled, shell and green sand moulded, was narrower.

The method also avoids some of the difficulties associated with the use of conventional chills and it is considered that, if the technique is found to be practical in the commercial foundry, it shows considerable promise as an aid in the attainment of premium quality in shell mould castings.

#### Effect of Test Bar Variables on the Mechanical Properties of Magnesium Casting Alloys

The effect of machining on the tensile properties of magnesium alloy test bars was investigated. Although some of the differences in property values were found to be statistically significant, they were of the same order as differences that were observed between melts of the same composition. The results illustrated that the mechanical properties obtained from substandard-size test bars may be significantly different from those obtained on standard test bars.

The study showed that results obtained on round test bars were significantly higher than those obtained on flat test bars cut from the same parts of the castings.

A comparison of 0.1% and 0.2% yield strength values, obtained on test bars of various magnesium alloys, showed that the linear relationship between these two values is different for most of the alloys and tempers investigated.

Similarly, linear relationships between the elongation values used in North America (4D), in Great Britain (3.5D) and in Continental Europe (5D) were found for the alloys investigated.

#### Effect of Wall Thickness on Tensile Properties of Mg-Al-Zn Alloy Castings

It has been shown in an earlier investigation that, under favourable solidification conditions (e.g., in premium-quality castings), high mechanical properties may be obtained regardless of wall thickness. In commercial castings, however, it is not always possible (or profitable) to avoid some reduction of mechanical properties in heavier sections of the casting. Section thickness per se has, of course, no influence on the properties, but it changes the solidification conditions, which in turn affect alloy structure (grain size, dendritic cell size, alloy constituents) and distribution (size, shape) of microporosity.

The present investigation was carried out to establish the effect of section thickness on properties of AZ80, AZ91 and AZ92 alloys in various temper conditions. Correlation of tensile properties with section thickness, grain size, surface-to-volume ratio, cooling rate and solidification time were determined. It was found that tensile properties decrease gradually with decreasing surface-to-volume ratio (decreasing cooling rate) until this ratio drops to about 3 (cooling rate below 30 C min or 54 F min):

beyond this, tensile properties drop more rapidly.

#### Foundry Characteristics of Copper Alloys

Continuing the study on the hot tearing of copper alloys, thermal analysis of representative alloys and metallographic examination of the test bars from all the alloys have been completed. An extensive literature survey has been carried out to correlate the present work with that conducted by others, and to examine more thoroughly the mode of freezing of the various alloys under conditions of non-equilibrium such as are encountered in actual castings.

From this work, it is suggested that the strain rate theory of hot tearing as proposed by Pellini offers the simplest explanation of the observed phenomena in copper alloys. However, the present work shows that, at least with the copper-tin alloys, the presence of a liquid lead-rich phase during solidification appears to have no adverse effect on the hot tearing characteristics, and indeed, when present in appreciable quantities, it has a beneficial effect.

An increase in zinc content, up to the point where the alloys begin to solidify as beta, appears to have a marked adverse effect on the hot tearing rating, and it is suggested that this is due to some modification of the properties of the primary alpha by zinc, which makes the alloys more susceptible to hot tearing. In the zinc-rich alpha alloys, lead may have an adverse effect on hot tearing, similar to the well known adverse effect on hot cracking during hot rolling in similar alloys.

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The thermal analysis data, when related to metallographic examination and constitution diagrams show that in many alloys containing even small amounts of tin, the freezing range is considerably wider than would be expected from other published work, and that the tin content has the predominant influence on the amount and type of the last liquid to freeze.

The type of last liquid to freeze would appear to be important in that lead-rich liquid in copper-tin alloys forms globules and has little effect on hot tearing. In zinc-rich alloys with primary alpha it occurs more at grain boundaries and may have a greater adverse effect. The tin-rich phase (which appears as delta in the room temperature microstructure) also tends to occur at grain boundaries, and at the intersections of grains, and the effect is dependent on the amount and composition. Finally, the last liquid to freeze in the silicon bronze alloy has a marked grain boundary distribution, and, as might be expected, the hot tear resistance of this material is very low.

Experimental work on the directional solidification of copper alloys has been completed, but the results have not yet been collated.

## POWDER METALLURGY

### Hypereutectic Aluminum-Silicon Alloys

Investigation of the production of hypereutectic aluminum-silicon alloys by powder metallurgy methods was extended and the effect of heat treatment on the hardness and tensile strength of aluminum-silicon alloys containing 25%, 35% and 45% Si was investigated. Brinell hardnesses of 65, 67 and 114, and ultimate tensile strengths of 33 kpsi, 32 kpsi and 40 kpsi were obtained for the extruded Al-25% Si, Al-35% Si and hot pressed Al-45% Si alloys respectively. Solution heat treating 535°C (1000°F) and ageing at 200°C (400°F) for times up to 4 hr reduced the hardnesses and did not improve the tensile strengths.

Wear resistance of these alloys was evaluated by measuring the weight loss incurred by a 1/8 in. diameter by 1/2 in. long pin of the alloy after the end of the pin had been in dry contact with a rotating cast iron disc under 1,000 psi loading and a speed of 200 in. per minute for 1 hr. Results in the form of weight loss were 13.2, 9.8 and 8.6 mg for 25% Si, 35% Si and 45% Si alloys respectively. This compares with 16.7 mg obtained for the 11.5% Si cast alloy.

### Nickel Powder

An investigation on the effect of blending nickel powders of different size fractions and particle shapes was made. This project included a study of the manner in which the particle size and shape relate to the apparent density and flow rate of

the powder and to the density and compressive strength of sintered compacts. Some work was also done to investigate the effect of sintering temperatures on these properties.

#### Powder Metallurgy Composite Materials

An exploratory investigation was made to produce and evaluate a number of composite materials of potential interest for use as coinage metal. The materials studied consisted of magnetic and non-magnetic base metals including nickel, stainless steel, nickel-5% silicon alloy and molybdenum, with each of which was incorporated varying amounts of silver as an insoluble, continuous second phase, or matrix. The results of this work indicated that, to produce an apparently homogenous, white composite material compatible with 800 fine silver coins in existing eddy current slug rejector devices, requires an unacceptably high proportion of silver and the product is difficult to fabricate. Further work is being done on other similar composites to complete the study.

## WELDING

### Welding of Structural Steels at Low Temperatures

This project is concerned with the metallurgical effects of welding structural steels at low temperatures. Conventional weldability tests on five different steels are being made in a cold room with temperatures in the range of 24°C to -51°C (75°F to -60°F).

Lehigh restrained-butt weld tests using E-6010 electrodes and a heat input of about 50,000 joules per inch established critical restraint levels of 3½ in. and 4½ in. for a 0.27% carbon steel and a 0.20% carbon steel respectively when tested at -51°C (-60°F), and critical restraint levels of 4½ in. and 5 in. respectively when tested at -34°C (-30°F).

Controlled thermal severity tests (CTS tests) have been used extensively in this work, sometimes with a 1/16 in. gap between the test plates to increase test severity (gap-CTS tests).

No cracking was obtained in either CTS or gap-CTS tests on the 0.20% carbon steel, with thermal severity number (TSN) of 12 at a test temperature of -51°C (-60°F), even with E-6010 electrodes and a low heat input of 22,400 joules per inch. Under similar conditions, cracking was obtained with the 0.27% carbon steel, the threshold heat input for cracking being 30,800 to 33,000 joules per inch for CTS tests and 54,200 to 70,400 joules per inch for gap-CTS tests.

CTS tests with TSN of 11 were made on a 2% nickel steel (A 203) at  $-51^{\circ}\text{C}$  ( $-60^{\circ}\text{F}$ ) using E-8016-C1 electrodes, and gave weld metal cracking with energy input in the range 35,000 to 37,800 joules per inch. No cracking occurred even with gap-CTS tests with energy input in excess of 50,000 joules per inch.

#### Automatic Vertical Welding of Steels

The study of electrogas or automatic vertical welding of steels was continued. Welds in 1 in. thick ASTM A373 structural steel, welded with filler wire containing 0.12% C, 1.15% Mn and 0.51% Si, were subjected to drop-weight testing. Unwelded plate was also tested to permit a comparison of the nil-ductility transition (NDT) temperature of plate and weld. The NDT temperature for the weld was  $-1^{\circ}\text{C}$  ( $30^{\circ}\text{F}$ ) compared with  $-7^{\circ}\text{C}$  ( $20^{\circ}\text{F}$ ) for the plate. Charpy V-notch impact values, corresponding to these NDT temperatures, were determined by tests to be 25 to 30 ft-lb at  $-1^{\circ}\text{C}$  ( $30^{\circ}\text{F}$ ) for the weld and 30 ft-lb at  $-7^{\circ}\text{C}$  ( $20^{\circ}\text{F}$ ) for the plate.

Considerable progress was made in an evaluation of the electrogas welding of 3/4 in. thick CSA G 40.8 Grade B steel containing 0.19% C, 1.38% Mn and 0.30% Si. Electrogas welds in this steel in both the as-rolled and normalized conditions were made employing the same filler wire as was used in welding the A373 steel. Joints 48 in. long were welded at a speed of about 2.2 in./min and with an arc energy input in the range 301,000 to 322,000 joules per in. No difficulty was encountered in producing welds having good quality, as shown by radiographic

examination and by satisfactory tensile and bend properties.

The major purpose of this study of electrogas welds in CSA G 40.8 Grade B steel was to evaluate the notch-ductility. One method of evaluation consists of determining Charpy V-notch impact values of specimens having notches, in separate series of tests, in various weld and heat-affected zone locations. The values obtained in the welded joint compare favourably with those for the prime plate.

The notch-ductility of electrogas welds is also being evaluated by explosion-bulge testing. The as-rolled plate without a crack-starter bead showed no cracking at  $-75^{\circ}\text{C}$  ( $-103^{\circ}\text{F}$ ), the lowest temperature at which tests were conducted. The presence of an electrogas weld raised the transition temperature to  $-42^{\circ}\text{C}$  ( $-43^{\circ}\text{F}$ ) though not as much as a crack-starter bead. With the latter, the transition temperature was about  $-5^{\circ}\text{C}$  ( $23^{\circ}\text{F}$ ) whether an electrogas weld was present or not. Similar though smaller effects were found when normalized plates were used, and the transition temperatures were generally lower.

#### Welding of Bronzes

Some previously reported experimental work devoted to the development of a suitable welding method for the repair of leaded bronze and gun-metal castings, resulted in the formulation of a new phosphor bronze (0.2% P) filler wire with added silicon (0.30% Si). Further work has been undertaken, including high-speed cinematographic recording of arc phenomena, designed to elucidate the effect of this addition.

The results have been analysed and the following conclusions have been drawn:-

1. The addition of silicon to the phosphor-bronze wire for the metal inert-gas welding process is necessary in order to deoxidize the weld pool and to stabilize the arc.

2. Welding tests have shown that, in some cases, the addition of silicon could reduce porosity to an acceptable level, even with rather high lead content (2.57%) in the base metal.

3. With added silicon, a quieter arc was produced, and spatter was almost negligible. Without silicon additions, spatter losses can vary between 15 and 30%.

4. Wetting and penetration were improved by the addition of silicon.

5. It is believed that silicon added to a nominal phosphor bronze composition reduces oxide formation on the molten pool, thus lowering cathodic resistance.

#### Welding of New Canadian Structural Steels

This project is concerned with the weldability testing of some new Canadian structural steels: CSA G 40.8 Grade A with 0.22% C max and 0.8 - 1.50 Mn, CSA G 40.8 Grade B

with 0.20% C max, 0.8 - 1.50 Mn and 0.35% Si max, and CSA G 40.12 with 0.22% C max, 0.8 - 1.50% Mn and 0.15 - 0.30% Si. A number of different weldability tests are being used for assessment.

Two samples of CSA G 40.8 Grade A steel have been compared with a Grade B steel of similar composition except for silicon content.

Double trithermal gap-CTS tests on one of the G 40.8 Grade A steels at 20°C (70°F) with TSN of 12, gave no cracking with energy input as low as 32,300 joules per inch. Even with TSN 11 and rutile electrodes, no cracking was produced with a heat input of 29,100 joules per inch. No cracking was found with the other G 40.8 Grade A steel in the cruciform test and the semi-cruciform test with an energy input of about 20,000 joules per inch and low-hydrogen electrodes.

The Grade B steel gave no cracking in CTS tests with heat input in the range 20,000 to 40,000 joules per inch, but gap-CTS tests, some of them double trithermal, gave severe cracking with energy input in the range 30,000 to 61,500 joules per inch. Cracking was reduced with an energy input of 63,000 joules per inch, and disappeared with an input of 66,000 joules per inch or above.

The Grade B steel is more susceptible to heat-affected zone cracking than the two G 40.8 Grade A steels, and the main composition difference is that the former contains 0.34% silicon compared with 0.03 - 0.04% silicon for the latter.

Further investigation is required, since previous findings suggest that silicon content does not have a pronounced adverse effect on weldability.

## SURFACE TREATMENT AND CORROSION

### Galvanized Coatings

Study of the deterioration, at elevated temperatures, of conventional galvanized coatings has revealed significant reaction effects, dependent largely on coating composition, but also on coating structure, and the time and temperature of heating.

Lead-containing coatings were distinguished by characteristic separation of the outer zinc layer resulting from bond destruction at the zinc-zeta interface. However, the usefulness of the separated layer as part of the coating is not necessarily lost since diffusion of zinc across the gap can continue if the layer is not induced to lift away by natural or other means. Lead-free coatings (0.001% Pb max), on the other hand, did not show separation at any stage and the zinc layer gradually disappeared to form iron-zinc alloy. Intimate contact was retained at all interfaces in these coatings, thus resulting in a higher zinc dissolution rate and more rapid transformation effects in the underlying iron-zinc alloy layers. However, special cases of cavity formation at the zinc-zeta interface have been observed in lead-free coatings when the coating geometry departs from a flat surface.

Coatings alloyed with tin, bismuth, thallium or indium, which are near neighbours of lead in the periodic table, behaved much like those containing lead.

In contrast, a series of ten other elements had no apparent effects and the coatings behaved in the same manner as those prepared with lead-free zinc.

Concurrent studies are in progress to compare the elevated temperature performance of commercial coatings on thick-wall tubing and angle products. Tests, to date, have confirmed prior findings on laboratory-prepared coatings. In all this work, it is shown that for long term heating exposure such coatings have a relatively low useful temperature limit.

#### Effect of Alloying Elements on the Corrosion of Chromium Stainless Steels

In a fundamental study of the effect of alloying additions upon corrosion behaviour, a number of heats based on AISI Type 430 stainless steel (17% chromium) have been prepared which contain additions of Si, V, Ge, Mo, Pd, Ta, W and Re, each at several levels. Each of these elements has good to excellent corrosion resistance in sulphuric and hydrochloric acids, and also has significant solubility in ferritic iron.

A preliminary assessment of the effect of the additions is being made by means of polarization measurements carried out in chloride-free and chloride-containing sulphuric acid solutions. The results, to date, indicate that chromium stainless steels containing each of Ge, Pd, and Re should show improved corrosion resistance. The largest positive effects were observed with steels containing palladium additions.

Environmental Cracking of High-Strength Materials

Work has been undertaken to determine the susceptibility of high-strength materials to cracking under stress, as a result of exposure to certain aggressive environments. In particular, an attempt is being made to determine the relationship between cracking due to hydrogen embrittlement and other cracking mechanisms, in some cases electro-chemical in nature, which may be termed stress-corrosion cracking.

Initially, techniques are being worked out whereby notched tensile specimens can be hydrogen-embrittled to varying degrees, and the varying degrees of embrittlement subsequently demonstrated by test methods. Varying the current densities and times of exposure have been tried during cathodic hydrogen charging. Varying the notch radius on a static tensile notched specimen is one method of varying the severity of this test. Modifications have been made to a standard cylindrical bend test specimen for a slow bend test method used to determine the degree of embrittlement by measuring the bend to failure.

Four different materials have been investigated to date, three of which are based on a proprietary Cr-Ni-Mo low alloy steel composition containing 0.25% C, 1.30% Mn and 1.50% Si. Two modifications were made, one containing low silicon and the other low manganese. The fourth material is a modified AISI 4320 composition. All were heat treated to a strength level of approximately 220,000 psi.

Evaluation by notched tensile testing and by slow bend testing have indicated that the proprietary steel and its low Mn modification probably have a greater degree of resistance to hydrogen embrittlement than the other two steels. The AISI 4320 steel is the least resistant of the four tested.

## MECHANICAL PROPERTIES

### Stress History and Spectrum Loading

This Division has been active in two international cooperative research programmes sponsored by the Organization for Economic Cooperation and Development. The first programme on the effect of cyclic stress history on the fatigue characteristics of a chromium-molybdenum steel has been completed by all participating organizations. The results have been evaluated statistically and the final OECD report on this programme should be issued within the current year.

The second programme on the effect of cyclic stress history on the fatigue characteristics of aluminum alloy CG42 (24S) is in progress. This programme has been revised on the basis of preliminary tests carried out in our laboratories and others in the U. K. A rigid specification for specimen form, preparation and tests has been drawn up and agreed upon by all the countries participating in the programme.

The initial phase of the revised programme consisted of the determination of the median endurance at three stress levels and the fatigue strength of  $30 \times 10^6$  cycles under rotating bending conditions. These tests have been completed by the Mines Branch and the U. S. Air Force Materials Laboratory at 3,000 and 10,000 cpm in constant bending moment R.R. Moore fatigue machines, and the results are being analyzed statistically.

The results from both laboratories show some evidence for the existence of a speed effect.

#### Atmospheric Environment and Fatigue

The study of the effect of environmental relative humidity on the fatigue behaviour of 57S-H34 aluminum alloy has been continued. The results obtained from pulsating tension tests at various stresses in environments of high, intermediate and low humidity have been analyzed statistically. In high humidity, the fatigue endurances show a pronounced bimodal distribution and the S/N diagram thus shows a normal curve with a second, approximately parallel, portion present at lower stresses. The secondary curve does not replace the primary curve, as it is not present at the highest or lowest test stresses used. A single curve only is found for tests carried out in a low humidity environment, while, for intermediate humidity, an apparently single curve of anomalous slope was found.

A similar statistical analysis of results from test pieces stored in various environments prior to testing in high humidity has shown significant variations in both the mean endurances and the standard deviations. Two important conclusions drawn were that test pieces stable with respect to the testing environment showed no bimodal distribution of results, and that the scatter from such tests is very low. On the basis of the data obtained it has been suggested that the environmental effect is due to variations in cracking of the oxide film due to property differences of films formed in differing environments.

Qualitative support for this hypothesis has been obtained by tests conducted using specimens with oxide films of various thicknesses produced by low voltage anodic oxidation.

#### Brittle Fracture

The conditions under which steel fails in a brittle manner are under continuing investigation. One of the common methods of studying this phenomenon is the Standard Oil Development(S.O.D.) test. In this test a notched test piece is loaded in tension to a pre-determined load level at a pre-determined temperature. This temperature will usually be below room temperature. While under tension the test specimen is impacted in the notch by a wedge shaped projectile. This impacting may, or may not, produce a crack. If a crack is produced, it may grow for a short distance and then stop, or it may continue to grow across the specimen causing failure by fracture.

A continuing part of this programme has been the determination of the relationship between test piece history and the ductile-brittle transition temperature (d.b.t.t.). The results of this programme to date, using Canadian Grade B steel have given d.b.t.t.'s between  $-83^{\circ}\text{C}$  ( $-117^{\circ}\text{F}$ ) and  $-1^{\circ}\text{C}$  ( $30^{\circ}\text{F}$ ) depending on the prior history of the material being tested.

Further tests have been carried out on Canadian Grade B steel to examine the relationship between the length of fatigue cracks and the fatigue hardened areas associated with these cracks. It would appear from the results of these tests that there is no simple proportional relationship between the extent

of the fatigue hardened area associated with a crack and its length. For example, the fatigue hardened area of a 0.040 in. long crack extended 0.020 in. from its root and 0.056 in. from the root of a crack 0.160 in. long.

Fractured surfaces of Canadian Grade B steel and 0.02% Nb steel, obtained by S.O.D. and notch tensile tests were examined and shear lip thickness and reduction in thickness values along the fracture length were obtained. The results are summarized in the following paragraphs.

The amount of ductility at fracture initiation in the S.O.D. test decreases with decreasing temperature. When fracture occurred at or below the d.b.t.t. the thickness of the fibrous zone at fracture initiation was much less than the shear lip thickness. In specimens containing sub-grain boundaries, these boundaries do not hinder crack propagation; however, grain boundaries can act as barriers.

Fracture initiation is more difficult than fracture propagation. If, by some transient means, a running crack can be initiated at room temperature above the ductile-brittle transition temperature, these cracks may continue to propagate even though severity of stress conditions decreases. However, in the brittle part of the fracture, the amount of ductility increases and the shear lip thickness increases.

In notch tensile tests on as-received un-fatigued material, the fibrous zone at fracture initiation is much thicker than in the S.O.D. test and is comparable to the shear lip

thickness, thus indicating the effect of strain rate. Much reduced fibrous zone widths are observed in fatigued specimens in S.O.D. tests and in notch tension tests when these are made below the d.b.t.t. It is considered quite feasible that the fatigued structure at the crack root is more brittle than the matrix and is a strong contributor, together with the small crack tip radius, to the increased d.b.t.t. of fatigued specimens.

#### Fatigue Crack Propagation

A research programme to examine the influence of such factors as metal composition, loading conditions, etc., on fatigue crack propagation has been under way for several years. A study of the rate of fatigue crack propagation in centre-notched flat specimens of mild steel, with and without niobium, has been completed this year.

It was found that at a 99% confidence level the addition of 0.02% niobium was detrimental to the fatigue tolerance of the steels tested and also caused an increase in the rate of propagation. The data were analyzed with respect to the generalized expression for crack growth rate

$$\frac{dL}{dN} = C (\text{stress range})^m (\text{crack length})^n$$

The constant "C" was found to depend on both the material and the mean stress. This is at variance with the opinions of other workers in the field who claim that the constant "C" is dependent only upon the material.

## DESIGN AND SERVICE EVALUATION

### Copper-Nickel Alloy Steels

A 10-ton heat of a copper-nickel alloy steel, developed by this Division, has been used for extensive evaluation tests.

Nil ductility transition tests were carried out on specimens treated to yield strengths ranging from 106,000 to 134,000 psi. The corresponding temperatures for nil ductility transition were between  $-110^{\circ}\text{C}$  ( $-166^{\circ}\text{F}$ ) and  $-60^{\circ}\text{C}$  ( $-76^{\circ}\text{F}$ ).

Ductile-brittle transition temperatures have been determined by explosive bulge tests on quenched and tempered prime plates 20 in. square by 1-1/8 in. thick with crack starter weld beads. When heat treated to a yield strength of 158,000 psi the ductile-brittle transition temperature was between  $-40$  and  $-48^{\circ}\text{C}$  ( $-40$  and  $-55^{\circ}\text{F}$ ). When heat treated to a yield strength of 114,000 psi the ductile-brittle transition temperature was  $-43$  to  $-51^{\circ}\text{C}$  ( $-45$  to  $-60^{\circ}\text{F}$ ).

Weldability studies have shown this steel to have good weldability at a yield strength of 114,000 psi. Heat affected zone cracking tests were carried out and included cruciform and restrained double-fillet tests on  $\frac{1}{2}$  in. plate, and CTS tests on 1 in. plate. No cracking occurred in any of these assemblies when welded with E-11018 electrodes having a coating moisture content of 0.17%. When CTS tests were welded with E-11018 electrodes having a coating moisture content of 0.7%, moderate heat-affected zone cracking occurred in one of the six test welds.

In an attempt to increase the severity, a CTS test was welded with E-6012 electrodes having a coating moisture content of about 5% and with energy input values of 35,000 to 38,000 joules per inch. No cracking was found.

Butt joints were welded in  $\frac{1}{2}$  in. plate, using the consumable-wire gas-shielded arc process. The procedure followed was the same as recommended for welding Hy-80 steel. Radiographically sound welds were produced. Average transverse tensile properties were 127 kpsi UTS, 122 kpsi yield strength, and 16% elongation in 2 in. Weldments for explosion bulge testing will be prepared.

#### Alloy Steel Chain

As a result of work carried out for the Alloy Steel Chain Cable Committee, the Department of Transport has replaced its  $1\frac{1}{2}$  in. to  $1\frac{1}{4}$  in. diameter mild steel buoy chains with 1 in. diameter low alloy steel chain for service on both east and west coasts. Some of these low alloy mooring chains have already been in service for two years. An examination of these chains after two years, has confirmed the tests originally carried out in the Bay of Fundy. They are giving equally as good service as the heavier mild steel chains that they replaced. Indications are that wear will still be a problem with the low alloy steel chains since the hardness is not much higher than for the mild steel chain.

A new copper-nickel steel alloy much harder and stronger than the low alloy steel referred to above has been developed by this Division. Due to its higher strength, this new steel should permit the use of 5/8 in. diameter chain with the same load carrying ability of the 1 in. diameter low alloy steel chain. It should also have superior wear resistance due to its increased hardness. An entire mooring (270 ft) of 5/8 in. diameter chain will be produced in this new steel for full scale testing in the Bay of Fundy.

#### Ultra-High-Strength Steels

A study of the mechanical properties of selected alloy steels at the 200-250 kpsi strength level is continuing in connection with the development of the R.C.N. hydrofoil vessel. The objective of this work is to evaluate this class of material for hydrofoil fins, and also to provide design data for this application.

A value of 105 kpsi inches<sup>1/2</sup> has been determined for the average plane strain fracture toughness of 18% nickel (250) maraging steel. Critical flaw sizes for this alloy for the probable conditions of hydrofoil operation were calculated as a guide to non-destructive test specification.

Similar tests were made on laboratory produced 12% nickel (200) maraging steel and commercially produced 17-4PH stainless steel. The 12% nickel maraging steel showed some superiority over the stainless steel. However, it will not be possible to measure true plane strain fracture toughness of these steels until the testing parameters are modified.

Fracture toughness was also investigated by means of standard and pre-cracked Charpy impact tests on 12% nickel maraging steel, commercial HP94 and 17-4PH steel. Air melted and vacuum arc re-melted 12% nickel steels were compared but no significant differences could be found.

The effect of shot peening and sand blast on the fatigue strengths of 18% nickel (250) maraging steel was examined. Shot peening with or without sand blasting superimposed resulted in the highest fatigue values, whereas shot blasting alone produced only a slight improvement.

The welding of the 18% nickel maraging steels by the metal-arc inert-gas (MIG) welding process has also been the subject of intensive investigation since it has been necessary to identify the weldability characteristics of steels of this type as part of the over all evaluation programme for hydrofoil fin material.

Work during the past year has identified a number of problem areas that can occur in the MIG welding of the 18% nickel maraging steels (250,000 psi yield strength). It has been found that unless great care is observed in attempting to remedy some of these problems other troubles will be introduced. For example, steps taken to remedy arc blow can introduce lack of inter-bead fusion. MIG welding with an alternating current source using specially treated filler wire was found to be the most satisfactory in respect of metal transfer and arc stability. However, welds produced by this technique resulted in joints showing some porosity and lack of fusion.

The welded joints gave evidence of poor resistance to crack propagation. This can be attributed to a combination of low impact toughness and ductility of the weld metal itself and possible low ductility in the heat affected zone. No means of improving the toughness of the welded joint was discovered short of annealing for one hour at 815°C (1500°F) after welding.

#### Quebec North Shore and Labrador Rail Samples

A study of the metallurgical characteristics of rail steel used in the Quebec North Shore and Labrador Railway between 1951 and 1963 has been undertaken in an effort to identify some relationship between service life and some metallurgical factors. No such relationship has been found.

## TESTING PROCEDURES AND STANDARDS

### Evaluation of Test Methods for Refractory Metals

The Division continued to participate in the cooperative programme undertaken by the Structures and Materials Panel of AGARD to evaluate test methods for refractory metals and to develop specifications for the testing of these materials. As part of the refractory metals round-robin testing programme initiated by this Panel, a series of tensile tests on TZM molybdenum alloy sheet material has been completed and a report on the results prepared. These tests were performed at temperatures ranging from ambient to 1800°C (3272°F).

Work on unalloyed refractory sheet materials has been resumed and tests are now being conducted on tantalum sheet samples. For these samples the temperature range is being extended to a maximum of 2100°C (3812°F) and, as in the past, the elevated temperature tests are being performed in a vacuum furnace.

### Certification of Industrial Radiographers

The Physical Metallurgy Division acts as the examining authority for the certification of industrial radiographers throughout Canada. Senior Grade candidates for certification must pass a written and practical test, whereas Junior Grade candidates must pass a written test only. The Department of National Health and Welfare supervises the radiation safety portion of the examinations.

In 1965, seventy candidates were successful in the Junior Grade tests and thirty-two in the Senior Grade tests.

Ultrasonic Determination of Internal Defects in Metals

Ultrasonic techniques are being developed for the detection and location of internal defects in very thick steel sections and also in welds.

Small defects are difficult to detect in steel sections over 4 inches thick using radiographic techniques unless extremely powerful X-ray equipment is available. Ultrasonic testing can be usefully applied to the detection of internal flaws in very thick sections of steel and other metals. Accordingly, a program has been organized to develop familiarization with the application of ultrasonic techniques to this type of testing. Preliminary work has resulted in the development of a suitable medium to couple the transducer to rough surfaces, such as those found on unmachined castings. Using this coupling medium good correlation has been achieved between the location of defects using ultrasonic detection and its actual position after sectioning.

Underbead cracks in welds have been difficult to locate by reflected wave ultrasonic techniques because the defect may occur in a plane parallel to the surface of the material being examined. A study of a dual probe through-transmission technique is under way using test welds of controlled thermal severity. Lead zirconate probes, which are smaller in size and more sensitive, are being substituted for the larger quartz probes.

The defect size is calibrated by a standard test hole and is compared to the size of the indication viewed on the screen of the instrument. The actual size is then found by sectioning and using the magnetic particle test method.

## PHYSICS OF MELTING AND SOLIDIFICATION

### Properties of Liquid Metals

#### a) Viscosity of Lead and Lead-Tin Alloys

The influence of tin on the viscosity of liquid lead throughout the complete range of composition of the alloy system has been studied. Logarithmic decrement-temperature curves have been obtained which show that the addition of the solute to the solvent liquid causes a gradual change in viscosity, which is always in the direction of the viscosity of the pure solute, as was previously shown in the zinc alloy programme.

#### b) Density of Lead and Lead-Tin Alloys

The density of the same lead-tin alloys was also studied. It was found that a linear relationship exists between the liquid density and temperature. The relationship between atom per cent tin and the computed molar volumes shows excellent linearity. Thus, the lead-tin system displays a high degree of ideality and a mixture of the two pure components does not display a marked volume change when compared with the separate components in the pure state.

Density measurements on pure indium are under way.

#### c) Surface Tension

1) Lead - The surface tension of 99.9999%, 99.999% and 99.997% Pb has been determined in a closed isothermal cell over a temperature range from the melting point of lead, 327.5°C, to 567°C (620°F to 1050°F).

At the melting point, the surface tension of 99.9999% Pb was found to be about 1% greater than that of lead of 99.997 purity. The temperature coefficient of the surface tension of lead is negative. The data for the 99.9999% and 99.999% grades are best represented by two straight lines with gradients between  $-0.11$  and  $-0.15$  dynes/cm/°C separated by a plateau between  $420$  and  $490^{\circ}\text{C}$  ( $790$  and  $915^{\circ}\text{F}$ ). However, this plateau is much less marked in the 99.997% grade. The main difference between the surface tension-temperature curves of the 99.9999% and 99.999% grades is that at all temperatures within the range studied the surface tension of the purer grade is about  $3.5$  dynes/cm higher than that of the lower grade.

Continuous vaporization during surface tension measurements depresses the lead surface tension results by about  $17$  dynes/cm and makes the slope slightly less negative.

2) Tin - The surface tension of 99.9999% Sn has been measured from  $560^{\circ}\text{C}$  ( $1040^{\circ}\text{F}$ ) down to supercooled Sn at  $200^{\circ}\text{C}$  ( $390^{\circ}\text{F}$ ). The data show a negative temperature coefficient of surface tension represented by a straight line with a slope of about  $-0.086$  dynes/cm. The results determined on ascending and descending temperature sequences in hydrogen and in helium agree within  $5$  dynes/cm.

Under similar non-equilibrium vaporizing conditions as those imposed on lead, the surface tension of tin below  $460^{\circ}\text{C}$  ( $860^{\circ}\text{F}$ ) was found to be depressed from  $7$  to  $15$  dynes/cm compared with the equilibrium surface tension.

Between 460°C (860°F) and the upper temperature limit of experiment (520°C (968°F)), the surface tension becomes independent of temperature.

3) Zinc - The positive temperature coefficient of the surface tension of zinc earlier determined in hydrogen, was confirmed by measurements in helium.

4) Lead-Tin Alloys - Work has begun on the surface tension of the lead-tin system. The coefficients of surface tension have been determined for alloys containing 12.14% Pb, and nominally 25% Pb, and further work in this system is proceeding.

#### Progressive Solidification from the Melt

##### a) Diffusion in Liquid Alloys

To interpret the results of a continuing study of impurity sub-structures formed during solidification of dilute silver in tin alloys, reliable values for the diffusion coefficient of silver in liquid tin were needed. These have been determined, using the capillary-bath method. The value obtained over the temperature range 250° to 500°C (480° to 930°F) was  $D = 2.5 \times 10^{-4} \exp(-2480/KT) \text{ cm}^2/\text{sec}$ .

The work is now being extended to a study of diffusion in liquid lead-tin alloys.

##### b) Grain Structure and Solute Segregation in an Undercooled Bismuth-Silver Alloy

Small ingots of 100 ppm Ag<sup>110</sup> in bismuth have been solidified at undercoolings of up to 70°C (126°F). Autoradiographs were taken from sectioned surfaces, and the observed

solute segregation correlated with the grain structure. It was found that as the undercooling was increased, the grain structure changed from equiaxed to columnar and then to large grains containing subgrains, with a wide scatter of orientation.

c) Tin-Thallium Alloys

A series of alloy single crystals were solidified at known growth rates and temperature gradients and the onset of corrugation segregation examined by autoradiographic techniques.

The general appearance and density of the microsegregation associated with the corrugation sub-structure was determined in the concentration range 0.001 to 1% of Tl at growth rates in the range of 7 to 30 cm/hr.

## STRUCTURES AND TRANSFORMATIONS

### Fatigue of Copper Single Crystals

The examination of the structure of persistent slip bands in copper single crystals fatigued by plane bending has been continued.

Using transmission electron microscopy techniques, it has been shown that persistent slip bands are made up of elongated cells near the specimen surface. The cell walls are small angle boundaries, with a rotation angle of less than a degree of arc. The longer cell walls are pure twist boundaries made up of dislocations lying in the primary slip plane.

Tapered sections of specimens fatigued by both plane bending and push-pull show that extrusions and intrusions are both present in the persistent slip bands contrary to the results of Boettner and co-workers who reported only extrusions were formed.

Present results have indicated that there is a correlation between the extrusions and intrusions observed on the specimen surface of fatigued specimens and the elongated cell structure below the surface.

The cell structure in the persistent slip bands appears to be highly stable during annealing. No significant change in the cell structure was observed when the specimens were annealed for one hour at 430°C (805°F).

The matrix structure, consisting of clumps of tangled dislocations disappeared during this annealing treatment.

Determination of Surface Atom Positions  
by Ion Bombardment Patterns

Ejection patterns produced by the ion bombardment of single crystals contain spots other than those produced by ejection along close or near-close-packed rows. It is believed that these spots were built up from atoms which occupied non-normal positions on certain surfaces at the time of receiving glancing impact from within the crystal along a close-packed row of atoms.

These extra spots are clearest with face-centered-cubic metals and it is of some theoretical interest to see if any relationship exists between the frequency of occurrence of these surface atoms in non-normal lattice positions and the positions of f.c.c. elements in a group in the periodic table. Data have been obtained from the Group I metals, copper, silver and gold.

Preliminary results indicate that the apparent proportion of atoms in non-normal lattice positions is not dependent on ion flux density and probably not on ion energy. However, recently published data, obtained by low energy electron diffraction, indicates that even partial monolayers of absorbed gases can bring about systematic rearrangement of metal atoms on the surfaces of some metals. Thus, experiments must now be done under much more ideal conditions.

To this end an improved vacuum system has been built, consisting of refrigerated sorption pumps, an ion pump, all of which are free of the hydrocarbon or silicone oils contained in mechanical and diffusion pumps.

Apparatus for measuring the rate of sputtering of single crystals, by means of quartz oscillating collectors, has been assembled and is now undergoing tests.

#### The Use of Ion Bombardment Patterns for Introducing Crystallography to Non-Crystallographers

Students in many fields, including metallurgy and ceramics, require at least a rudimentary knowledge of crystallography so that they may become aware of the importance of crystallographic factors on their problems. To supplement a brief theoretical approach, they should perform a few simple experiments in laboratory courses so that they may at least obtain a working familiarity with a simple crystal lattice. Ion bombardment experiments seem particularly suitable for this purpose because ejection patterns from single crystals indicate directions, rather than reflections as in diffraction techniques, and thus the patterns are very simple to interpret. Moreover, when the patterns are obtained on hemispherical collectors, such as ping-pong balls, from specimens mounted at the centre of curvature of the collector, they may be used to directly produce stereographic or gnomonic projections in an optical device.

In addition to the simple ion bombardment camera, ancillary equipment and techniques have been developed which

because of their simplicity should find a place in the introduction of crystallography to non-crystallographers, particularly in physics departments with a limited budget.

### The Plastic Deformation of Metal Crystals

#### a) The Deformation of Lead

Measurements have been made of the critical resolved shear stress of lead single crystals over the temperature range  $-195^{\circ}\text{C}$  to  $275^{\circ}\text{C}$  ( $-320^{\circ}\text{F}$  to  $530^{\circ}\text{F}$ ). It was found that the shear stress could be reduced, with less scatter, by annealing the specimens in-situ. In addition, a small amount of work hardening could be removed by annealing, thus enabling the same crystal to be used for several shear stress determinations.

The results indicated that there was no significant difference in the shear stress for 99.9999% and 99.999% purity materials nor for crystal grown in a good vacuum, poor vacuum, or in air. Alloy crystals containing 1% Sn in Pb, after annealing, had a shear stress approximately double that of pure lead at  $-195^{\circ}\text{C}$  ( $-320^{\circ}\text{F}$ ), this difference decreasing with increasing temperature.

Measurements of flow stress ratio of 0.1% and 1.0% Sn in Pb crystals gave results essentially similar to the high purity lead.

Observations were also made on the cooling rate of the specimens during temperature cycling measurements, and the

amount of static recovery occurring during these measurements.

b) Slip Band Continuity Across  
Grain Boundaries in Aluminum

Two series of melt-grown aluminum bicrystals were electro-polished and pulled in tension. The distribution of slip bands at the grain boundaries was examined, and analyses made in terms of stress activation. The applicability of Stroh's analysis for the stresses near the head of a dislocation pile-up and of the Livingston-Chalmers analysis for the nucleation of secondary slip systems at a grain boundary was examined. Of the two, the Stroh analysis fitted the observations on slip band continuity much better, indicating that the stress fields from individual slip bands are the controlling factors.

Precision Lattice Parameter Measurements

Dispersion, Lorentz and Polarization Corrections  
in the Determination of Lattice Strain

The effect of dispersion, and the Lorentz and polarization factors on the mean lattice parameter and the residual lattice strain and domain size distributions determined from X-ray diffraction lines has been examined on standard specimens of low residual strain. The distortion due to the dispersion effect becomes largest in the high  $2\theta$  region where the majority of systematic errors become small, and the probable accuracy of lattice parameter measurement is highest. The effect of dispersion on the line centroid determination becomes catastrophic for  $2\theta \approx 165^\circ$ . The effect on the line centres determined by Fourier

methods was found to be of the second order, and a correction formula has been derived which can be used to correct both chord centres of the measured distribution and the angular displacement of the characteristic wavelength distribution determined by Fourier unfolding. The unfolding, carried out using the CuK $\alpha$  distribution determined by Parratt showed that the measured chord centres and the angular displacement values agreed within the estimated error in angular measurement of  $.003^\circ$  in  $2\theta$ .

PUBLICATIONS

1. R. L. Cunningham and Joyce Ng-Yelim - "Simplified Apparatus and Technique for the Determination of Crystal Orientation by Ion Bombardment" - Rev. Sci. Instruments 36(1), 54-56 (Jan. 1965).

The ion bombardment camera, designed specifically for orientation determinations, is machined from clear acrylic plastic and incorporates an ion source of the glow discharge type. A plastic orienting sphere, illuminated from within, has engraved on it the low index directions of cubic crystals. Hemispherical collectors, bearing ejection patterns are positioned on the sphere and the orientation of the crystals may then be immediately observed.

2. G. P. Contractor - "Some Properties of Plain Carbon Steels Containing Uranium" - Iron and Steel 38(1), 22-29 (Jan. 1965).

Medium- and low-carbon steels, modified with uranium, were studied in order to evaluate the influence of uranium on forgeability, mechanical properties, response to heat treatment and microstructure. Uranium contents ranged up to 0.70%. The medium-carbon steels are hot short if the uranium content exceeds about 0.35%. More uranium can be tolerated in low carbon steels. The hot shortness is caused by the formation of the intergranular phase  $UFe_2$ . Critical transformation temperatures, Jominy hardenability, and resistance to tempering are not significantly affected by the presence of uranium. Similarly, uranium has little or no effect on the tensile properties of carbon steels. The addition of uranium has a favourable effect on the stress-rupture characteristics and some beneficial effect on fatigue properties. However, more results are required for verification. In the steels investigated, it seems that most of the uranium carbide combines to form complex inclusions of U (O, C, N) or U (C, N), rich in oxygen or nitrogen or both. Above 0.35% U, uranium carbide is believed to occur as discrete spheroidal or dot particles which are stable at heat treating temperatures.

3. H. R. Thresh - "The Viscosity of Liquid Zinc by Oscillating a Cylindrical Vessel" - Trans. Met. Soc. AIME 233, 79-88 (Jan. 1965).

An oscillational viscometer has been constructed to measure the viscosity of liquid metals and alloys to 800°C. An enclosed cylindrical interface surrounds the molten sample avoiding the free surface condition found in many previous measurements.

Standardization of the apparatus with mercury has verified the use of Roscoe's formula in the calculation of the viscosity. Operation of the apparatus at higher temperatures was also checked using molten lead. Extensive measurements on five different samples of zinc, of not less than 99.99% purity, indicate that (i) impurities at this level do not influence the viscosity and (ii) the apparatus is capable of giving reproducible data. The variation of the viscosity  $\eta$  with absolute temperature  $T$  is adequately expressed by Andrade's exponential relationship  $\eta V^{1/3} = Ae^{C/VT}$ , where  $A$  and  $C$  are constants and  $V$  is the specific volume of the liquid. The values of  $A$  and  $C$  are given as  $2.485 \times 10^{-3}$  and  $20.78$ ;  $2.444 \times 10^{-3}$  and  $88.79$ ; and  $2.169 \times 10^{-3}$  and  $234.8$ , respectively, for mercury, lead, and zinc. The error of measurement is assessed to be about 1%. Prefreezing phenomena in the vicinity of the freezing point of the zinc samples were found to be absent.

4. K. Winterton - "New Materials for Welding" - Can. Welder and Fabricator 55(12), 12-14 (Dec. 1965); 56(1), 12-14 (Jan. 1965).

Increasing interest is shown in quenched and tempered steels for civil construction. The tough high-strength maraging steels now have several rivals. Precipitation hardening stainless steels are being used for aerospace application, notably in the form of honeycomb panels. The use of titanium is increasing. New alloys continue to appear with titanium and the other light metals. Nickel alloys are important for high-temperature applications. Inconel X is used extensively in the X-15 manned rocket plane. The refractory metals, molybdenum, tungsten and niobium, begin to find applications for special purposes. The boost given to materials technology by the aerospace industry has brought many new welding problems. Welding techniques are rapidly being developed for the new materials.

5. H. H. Bleakney - "The Creep-Rupture Embrittlement of Copper" - Can. Met. Quarterly 4(1), 13-29 (Jan.-Mar. 1965).

The rate of embrittlement of both high-purity and tough-pitch copper and the metallographic appearance of selected samples of the broken test bars were determined. The results indicate that the creep-rupture embrittlement of both tough-pitch and high-purity copper is influenced by oxidation through a stress-corrosion mechanism and by internal oxidation of grain boundaries. The difference between tough-pitch and high-purity copper was explained on the basis of the relative influence of the two mechanisms in each material.

6. W. P. Campbell - "Recent Developments in Welding Processes" - Can. Welder and Fabricator 56(2), 12-16 (Feb. 1965).

Developments in welding processes have accelerated rapidly in recent years. In general, the trend has been to higher welding speeds and greater automation, or to specialized processes that make possible welding that could not have been done satisfactorily with earlier processes.

This paper describes briefly some of the newer welding processes, such as consumable-wire gas-shielded metal arc, plasma-arc, electroslag and electrogas, induction, electron-beam, resistance, ultrasonic, explosive, laser, and friction.

7. D. M. Fegredo - "The Fatigue of Zinc Single Crystals in Reversed Shear" - J. Inst. Metals 93, 189-195 (Feb. 1965).

S/N curves have been obtained in reversed shear for zinc crystals, fatigued on (0001) along  $[11\bar{2}0]$ , at 25 and  $-55^{\circ}\text{C}$ . At  $25^{\circ}\text{C}$  the fatigue behaviour appears to be conventional; at  $-55^{\circ}\text{C}$  the curve indicates a high stress-dependence for failure. Specimens, which were initially fatigued at  $-55^{\circ}\text{C}$ , were tested in unidirectional shear at  $-55$  and  $-196^{\circ}\text{C}$ . The crystals rapidly acquired a flow stress which was approximately equal to the peak fatigue stress, and which showed a low temperature-dependence. Recovery experiments indicated that a well-defined activation energy of recovery does not exist over a range of temperature for crystals fatigued at  $-55^{\circ}\text{C}$  for various numbers of cycles. A general discussion is given of the results. The fatigue machine used to test the specimens in reversed shear is described in an Appendix.

8. D. M. Fegredo - "The Effect of Quenching on the Yield Stress of Zinc Single Crystals" - J. Inst. Metals 93, 268-274 (Apr. 1965).

Zinc single crystals of varying orientation were quenched from  $408^{\circ}\text{C}$  into water at  $2^{\circ}\text{C}$  and tested in tension at  $-196$  and  $-55^{\circ}\text{C}$ . Increases in yield stress of up to an order of magnitude were obtained. Annealing treatments of quenched crystals indicated that they possess greater thermal stability than crystals that are work-hardened or fatigue-hardened at  $-50^{\circ}\text{C}$ . A large temperature-dependence of flow stress was obtained only for unaged, quenched crystals. The results are discussed generally in terms of the effects of quenched-in vacancies.

9. A. Winterton - "The New Welding Processes" - Paper No. SP65-101, Creative Manufacturing Seminars, Am. Soc. Tool and Mfg. Eng., pp. 1-21 (Apr. 1965).

Fourteen new welding processes that have appeared in the last decade have been briefly reviewed. Four of these, i.e. remote control TIG welding, short-circuiting MIG welding, arc spot welding and plasma welding show a logical connection in their development. The remainder, laser welding, electron-beam welding, ultrasonic welding, explosive welding, electroslag welding, electrogas welding, radio-frequency resistance welding, diffusion bonding and foil-butt seam welding tend to be individual and distinct.

An attempt has been made to cover briefly for each of these processes the principle of operations, the general characteristics, advantages and disadvantages, typical applications, and an indication of their technical importance at the present time.

10. D. E. Parsons, B. Wallace, V. Kennedy and C. W. Briggs - "Vacuum Degassing for Steel Castings" - Tech. Research Comm., Steel Founders' Soc. of Amer., Research Rep. No. 57 (May 1965).

The effect of vacuum treatment on chemical composition, mechanical properties, macrosegregation and gas content are discussed. The results of previous work on carbon steel are reviewed and new data are included pertaining to alloy steel castings of the AISI-8630, AISI-4330 grades. Vacuum deoxidation of silicon-killed and "open" steels is discussed and the application of this technique to castings production is illustrated.

11. A. K. Laing, R. K. Buhr, S. L. Gertsman - "Navigation Buoy Mooring Chains" - VIIIth Int. Conf. on Lighthouses and Other Aids to Navigation, Rome (May 1965).

A wide variety of steels has been tested as potential replacements of  $1\frac{1}{2}$  to  $1\frac{1}{2}$  inch (0.0317 to 0.038m) diameter mild steel or wrought iron chain heretofore used to moor large navigation buoys in salt water. As a consequence of the work, it was found that the main causes of chain deterioration in this application were pitting corrosion and inter-link and barrel wear.

It has been shown that a higher strength 1 inch (0.0254m) diameter chain can satisfactorily moor a buoy even though the weight of chain is reduced by one half. Most low alloy steel chains tested suffered from pitting corrosion. However, two steels were found which displayed more resistance to this type of corrosion.

One, called Stelcoloy, is a low alloy copper-bearing steel which is commercially available. This composition is currently being used by the Canadian Department of Transport in place of mild steel or wrought iron chain to moor their buoys.

It has been found to be more economical. The second steel is not produced commercially at the present time. It contains higher percentages of copper and nickel and can be heat treated to high strengths. Large scale tests of this alloy are planned on 5/8 inch (0.0159m) diameter chain. It has been established that the 5/8 inch (0.0159m) chain is strong enough for mooring buoys, with a further substantial weight saving and probable reduction in cost.

12. J. W. Meier and B. Lagowski - "Magnesium Casting Out-Performs Aluminum Forging in Mortar Base" - *Modern Metals* 21(5), 54-55 (June 1965).

Development of a sand-cast magnesium alloy mortar base was considered at the end of World War II, but the properties of the magnesium casting alloys then available were not sufficient to achieve the needed weight reduction at comparable strength. Unreliability of casting quality defeated the project.

Now, the high-strength alloys (ZK61, QE22, and ZQ64) and the use of premium quality casting methods has made possible the development of a cast magnesium medium mortar base which is 10% lighter than the standard aluminum forging and has up to 30% higher strength with twice the rigidity.

13. W. A. Pollard - "Long-Term Ageing Behaviour of Aluminium-10% Magnesium Casting Alloys" - *J. Inst. Metals* 93, 339-346 (June 1965).

The ageing and related phenomena concerning the aluminium-10% Mg casting alloy, at room temperature and up to 150°C, have been studied by tensile testing and metallographic examination over periods of up to six years. In alloys containing  $\sim 10.2\%$  magnesium, aged at room temperature, a slow but continuous increase in strength takes place, at least up to five years. However, at 50°C and above this age-hardening does not occur, but precipitation is observed, first at grain boundaries, with no change in properties, and then in Widmanstaetten form within grains, accompanied by rapid embrittlement. Reversion of the room-temperature-aged material occurs above 50°C. The results are discussed in terms of work on the aluminum-magnesium alloys and of recent theories of age-hardening.

14. A. J. Williams - "A Study of the Constitution of the Titanium-Rich Corner of the Titanium-Aluminum-Molybdenum System" - *Can. Met. Quarterly* 4(3), 181-203 (July-Sept. 1965).

The constitution of the titanium-rich corner of the titanium-aluminum-molybdenum system, based on a four-hour annealing time, has been investigated.

Vacuum heat-treatment and metallography have been used to determine the beta-transus on nine constant titanium sections of this system in the composition range of 0-25 wt % molybdenum and 0-15 wt % aluminum. Using these sections, together with published data on the titanium-aluminum and titanium-molybdenum binary diagrams, isothermal sections at 850, 900, 950 and 990°C (1560, 1650, 1740 and 1810°F) have been developed and tie-lines have been determined for these sections. Four discontinuities in the beta-transus surface have been discovered. These discontinuities appear to indicate the presence of four three-phase fields which are contiguous with two-phase fields in the titanium-aluminum binary diagram discovered by several other workers.

15. B. Lagowski and J. W. Meier - "Further Development of Magnesium-Zinc-Silver-Zirconium Casting Alloys" - Trans. AFS 73, 246-254 (1965); Modern Castings 48(1), 64-72 (July 1965).

In continuation of earlier work, additional casting alloys were developed in the magnesium-zinc-silver-zirconium system to achieve the optimum combination of mechanical properties and foundry characteristics. In particular, some work was directed to the production of thin-walled premium-quality castings.

The effects of section thickness, heavy chilling, and hot water quenching after solution treatment, on the mechanical properties of high-strength magnesium casting alloys were investigated. Results of a study of foundry characteristics, including hot tearing, fluidity (castability), susceptibility to microshrinkage, linear shrinkage and density, are reported.

It was found that alloy ZQ91-T6 shows great promise for applications in more complex, and especially in thin-walled castings.

16. G. W. J. Waldron - "A Study by Transmission Electron Microscopy of the Tensile and Fatigue Deformation of Aluminum-Magnesium Alloys" - Acta Met. 13, 897-906 (Aug. 1965).

The formation of a cell structure in aluminum deformed in tension was found to be inhibited by the addition of small amounts of magnesium and completely suppressed in a 7% magnesium alloy. In this material a high density of dislocations was observed to be present at failure. Suitably oriented segments of helical dislocations appear to glide and may act as dislocation sources during tensile deformation.

When aluminum-magnesium alloys are fatigued, clusters of jogs and loops form similar to those reported in other f.c.c. metals. In an alloy below the solid solubility limit, the clusters develop into a sub-structure at high

stresses; above the solid solubility limit a sub-structure is not observed. In this latter case, a stage of uniformly distributed loops and jogs is seen prior to cluster formation. The effect of the solute is to inhibit both cluster and sub-structure formation.

It is believed that the effect of the solute in tensile and fatigue deformation is to interfere with cross-slip and the absorption of point defects at dislocation lines.

17. W. A. Pollard - "Layer Porosity Effects in Aluminum-10% Mg Alloy Castings" - Modern Castings 48, 99-107 (Sept. 1965); Trans. AFS 73 (1965).

The effects of layer porosity on the tensile properties of CSA test bars (similar to the "Navy" bar) and of chilled plate castings of Al-10% Mg alloys were studied for two levels of purity and over a range of magnesium contents.

As previously found for Dow-type test bars, the layer porosity occurred in the gauge lengths of CSA test bars, causing low tensile properties when the magnesium content fell below a critical value (about 10% Mg).

In chilled plates it was shown that, if the layer porosity was aligned parallel to the applied stress, then its effect on tensile properties was comparatively slight; whereas, if the layers were at right angles to the applied stress, the alloy was seriously embrittled. A correlation was found between magnesium content and the distribution of layer porosity that was similar to but less pronounced than that previously observed on separately-cast test bars.

18. J. W. Meier - "Research on Premium-Quality Castings in Light Alloys" - Proc. 32nd Int. Foundry Congress, Warsaw, Poland, Paper 5, 19 pp. (Sept. 1965); Trans. AFS 73, 525-540 (1965).

Extensive research on the structure and properties of cast aluminum and magnesium alloys has been applied to foundry practice. The term "premium quality" is used to describe castings with reliably high mechanical properties and high integrity of the product, guaranteed by the foundry. In production, conventional equipment and manufacturing techniques may be used, but rigid control of metal purity, alloy composition, melt quality, solidification conditions and heat treatment is essential to achieve and maintain high reliability of properties in designated areas of the casting, which are graded according to design and service considerations. Examples of the excellent properties that have been obtained in aluminum and magnesium alloy castings are listed.

Considering currently achieved properties, it is believed that in the near future aluminum alloy castings with 70 kpsi (50 kg/mm<sup>2</sup>) UTS, 60 kpsi (42 kg/mm<sup>2</sup>) 0.2% YS and 10% El, and magnesium alloy castings with 60 kpsi (42 kg/mm<sup>2</sup>) UTS, 50 kpsi (35 kg/mm<sup>2</sup>) 0.2% YS and 10% El, will be developed.

19. K. G. Davis and P. Fryzuk - "Solute Mixing by Thermal Convection in Horizontal Rods of Molten Alloy" - Trans. Met. Soc. AIME 233, 1796-97 (Sept. 1965).

In an earlier investigation of solute distributions along unidirectionally solidified rods of dilute silver in tin alloys, it was concluded that, for rods of 2 mm diameter or larger, a very high degree of convective mixing takes place in the melt ahead of the advancing interface. Suggested causes for this mixing were (1) thermal convection, (2) convective currents set up by changes in fluid density associated with concentration gradients, and (3) currents set up by changes in volume during freezing. Evidence was presented that causes (2) and (3) were not primarily responsible for solute mixing. Further, convective mixing took place in horizontal rods with temperature gradients as low as 6°C per cm, putting doubt on the importance of thermal convection.

In the present work, convective mixing is examined in melts with known initial solute distributions. The absence of a moving solid-liquid interface eliminates convection caused by volume changes on solidification.

20. K. G. Davis and P. Fryzuk - "Diffusion of Silver in Liquid Tin" - Trans. Met. Soc. AIME 233, 1662-1664 (Sept. 1965).

The diffusivity of silver in liquid tin has been determined, using the capillary-reservoir technique, over the temperature range 250° to 500°C. The new value,  $D = 2.5 \times 10^{-4} \exp(-2480/RT)$  sq cm per sec, differs from that obtained by other workers in an earlier investigation. The analysis of data from the capillary-reservoir technique is discussed.

21. C. F. Dixon and H. M. Skelly - "Hypereutectic Aluminum-Silicon Alloys Produced by Powder Metallurgy Techniques" - Int. J. Powder Met. 1(4), 28-36 (Oct. 1965).

Three hypereutectic aluminum-silicon alloys containing 25%, 35% and 45% Si were prepared using powder metallurgy techniques. Pre-alloyed powders, which were produced by atomization of the molten alloys were fabricated by hot pressing and extruding to give material with density close to theoretical.

The main object of the investigation was to determine if atomization of the alloy would result in refinement of the primary silicon, and metallographic examination of the powders showed that this was accomplished, although additions made to the melts assisted in such refinement.

Raising the silicon content resulted in a decrease in coefficient of thermal expansion and an increase in wear resistance.

22. Y. L. Yao - "Thermodynamics of Parabolic Time Rate Law of Oxidation of Metals" - J. Chem. Phys. 43(9), 3050-3051 (Nov. 1965).

The Wagner equation for the isothermal oxidation of metals is re-established. One of the assumptions used in deriving the equation is removed.

23. K. G. Davis and P. Fryzuk - "Grain Structure and Solute Segregation in Bismuth Ingots Solidified From Undercooled Melts" - Trans. AIME 233, 1983-1986 (Nov. 1965).

A study has been made of the effect of undercooling on the grain structure and solute distribution in small ingots of pure bismuth and of a 100 ppm silver in bismuth alloy. Autoradiographic evidence shows that in undercooled melts a network of dendrites is formed throughout the melt immediately after nucleation. At large undercoolings the dendrites are very thin and closely spaced. The origin of the observed grain morphology can be explained on the basis of information about the mode of solidification revealed by the impurity substructure.

24. R. L. Cunningham and Joyce Ng-Yelim - "The Ejection of Atoms from Metallic Single Crystals" - Am. J. Phys. 33, 1064-69 (Dec. 1965).

The unique characteristics of experimental data obtained from the directional ejection of atoms from metallic single crystals subjected to ion bombardment are discussed. The advantages, for instructional purposes, are outlined. Simple experiments are described that are designed to give the beginner in crystallography a working familiarity with (1) crystal directions, (2) techniques for orientation determinations, and (3) anisotropic characteristics of crystals. All experiments described may be performed with simple and inexpensive equipment.

25. G. L. Montgomery, B. Wallace, V. Kennedy and D. Parsons - "Vacuum Degassing Tests on 4330, 8630, 18-8 Stainless and 1030 Cast Steels" - Joint SCIC-SFSA Project No. 53, Steel Castings Inst. of Canada, (1964), (previously unreported).

The macrostructure, gas content (hydrogen, nitrogen and oxygen), tensile ductility, fatigue (notched and unnotched endurance limits and impact-transition temperature relation), were determined for non-degassed, vacuum stream degassed, vacuum ladle degassed and vacuum cast steel.

The steel compositions investigated were 1030 carbon steel, 18-8 austenitic stainless steel and the 8630, 4330 AISI grades.

Significant reduction of gas content, refinement of macrostructure and improvement of tensile ductility was obtained in 4 in. section castings when final hydrogen content was reduced below 0.0015 wt % with final oxygen content in the range 0.0020 to 0.0040 wt %.

26. D. E. Parsons - "Vacuum Degassing and Feeding of Carbon Steel Castings" - AFS Exchange Paper to the Australian Assoc. of Foundry Inst., Trans. Inst. South Australian Foundrymen (Oct. 1965): (previously unreported).

This work, previously published by the American Foundrymen's Society, pertained to vacuum degassing and vacuum casting procedures for steel. The effect of vacuum treatment of liquid steel on solidification, feeding mechanical properties and composition of carbon steel in 1 in. and 4 in. sections was reported.

27. A. H. Gillieson, D. J. Reed, K. S. Milliken and M. J. Young - "X-Ray Spectrochemical Analysis of High-Temperature Alloys" Symposium on X-Ray and Optical Emission Analysis of High-Temperature Alloys - ASTM Special Tech. Publication No. 376, pp. 3-22 (1965).

The factors involved in the correction of experimental results in the X-ray fluorescence analysis of high-temperature alloys are described and discussed. An empirical method for the mathematical correction of such results is detailed and the degree of correction assessed.

RESEARCH REPORTS, INFORMATION CIRCULARS,  
TECHNICAL BULLETINS

- R 102 "The Effect of Some Test Bar Variables on the Mechanical Properties of Aluminum Alloys" by A. Couture and J. W. Meier.
- R 144 "Orientation Determinations of Crystals Using Ejection Patterns Resulting from Ion Bombardment" by R. L. Cunningham and Joyce Ng-Yelim.
- R 146 "Simplified Apparatus and Technique for the Determination of Crystal Orientation by Ion Bombardment" by R. L. Cunningham and Joyce Ng-Yelim.
- R 149 "Research on Premium-Quality Castings in Light Alloys" by J. W. Meier.
- R 150 "Investigations on Sand-Cast Aluminum Alloy Test Bars" by W. A. Pollard and J. W. Meier.
- R 151 "The Effect of Test Bar Variables on the Mechanical Properties of Magnesium Casting Alloys" by A. Couture and J. W. Meier.
- R 152 "Properties of Sand-Cast Magnesium Alloys - Part VI: Effect of Pouring Temperature and Holding Time" by A. Couture and J. W. Meier.
- R 153 "Properties of Sand-Cast Mg Alloys - Part VII: The Effect of Section Thickness on Tensile Properties of Mg-Al-Zn Alloys" by A. Couture and J. W. Meier.
- R 157 "Theory and Experiment in Methods for the Precision Measurement of Surface Tension" by D. W. G. White.
- R 158 "Theory and Experiment in Methods for the Precision Measurement of Viscosity" by H. R. Thresh.
- R 159 "Density of Molten Zinc and Some Zinc Alloys" by H.R. Thresh.
- R 160 "Surface Tension of Molten Zinc and Some Zinc Alloys" by D. W. G. White.
- R 161 "Properties of Sand-Cast Magnesium Alloys - Part VIII: Foundry Characteristics of Magnesium-Zinc-Silver-Zirconium Casting Alloys" by B. Lagowski and J. W. Meier.
- R 164 "The Hot-Tearing of Copper Alloys" by A. Couture and J. O. Edwards.
- R 166 "Corrosion Behaviour of Uranium-Bearing Resulphurized Chromium Stainless Steels" by G. J. Biefer and W.M. Crawford.

- R 167 "Corrosion Fatigue of Structural Metals in Mine Shaft Waters" by G. J. Biefer.
- R 169 "Viscosity of Molten Zinc and Zinc Alloys" by H.R. Thresh.
- IC 168 "The Notch Toughness of Ultra-High Strength Steels in Relation to Design Considerations" by R.C.A. Thurston.
- IC 176 "The Preparation of "As-Polished" Metallographic Finishes in Non-Ferrous Metals" by R.I. Hamilton and E.F. Connors.
- TB 58 "The Effect of Uranium Additions on the Corrosion Behaviour of AISI Type 430 Stainless Steel" by G. J. Biefer.
- TB 71 "Weldability of Titanium and Titanium Alloys" by K. Winterton.
- TB 72 "The Status of the Hydrogen Problem in Steel" by R.D. McDonald.
- TB 73 "Polarization Measurements on ASTM Type 6061-T6 Aluminum Alloy in Three Ontario Mine Shaft Waters" by G.J. Biefer.
- TB 74 "A Comparison of the Effects of Uranium and Molybdenum Alloying Additions on the Corrosion Resistance of AISI Type 430 Stainless Steel" by G.J. Biefer and J.G. Garrison.

PATENTS AVAILABLE FOR LICENSING (from 1961).

"Ion Bombardment Camera for Crystal Orientation Determination, R. L. Cunningham, Joyce Ng-Yelim, A. V. Grant and K. V. Gow.

"Method of Removing Aluminum Oxides from Aluminum Killed Steels", G.P. Contractor and R.K. Buhr.

"Free Machining Steels of Improved Transverse Mechanical Properties", G.P. Contractor, D.E.C. King and R.J. McClure.

"Corrosion Pitting and Tarnish Resistant Stainless Steel, W.A. Morgan, D.E.C. King and R.J. McClure.

"Use of Uranium as a Scavenging Agent and to Improve the Malleability of Copper and Nickel Alloys", J.O. Edwards and R. Thomson.

"Prevention of Gas Absorption in Metals", N.S. Spence, L. Badone, W.A. Morgan and R.D. McDonald.