VOLUME 22

Advances in Cryogenic Engineering

Proceedings of the
First International Cryogenic Materials Conference,
July 22-25, 1975, at Queen's University

K. D. TIMMERHAUS, R. P. REED, AND A. F. CLARK, Editors

a CRYOGENIC ENGINEERING CONFERENCE Publication

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Edited by

K. D. Timmerhaus
Engineering Research Center
University of Colorado
Boulder, Colorado

and

R. P. Reed and A. F. Clark
NBS Institute for Basic Standards
Boulder, Colorado

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FOREWORD

The First International Cryogenic Materials Conference (ICMC) provided a new forum for the presentation of low-temperature materials research. The conference, held in conjunction with the 1975 Cryogenic Engineering Conference, provided materials research personnel with excellent exposure to current developments in the cryogenics field and beneficial interactions with designers of cryogenic systems. Because of the large response to a late call for papers, the enthusiasm and encouragement at the meeting, and the wide spectrum and high quality of papers, the Second International Cryogenic Materials Conference is being planned along with the 1977 Cryogenic Engineering Conference for Boulder, Colorado, in the summer of 1977.

The success of the First International Cryogenic Materials Conference was certainly in large measure due to the excellent hospitality of our Canadian hosts, the Royal Military College of Canada and Queen's University in Kingston, Ontario. In particular, the efforts of A. C. Leonard and his staff ensured an excellent conference and a pleasant and memorable visit to Canada. The Cryogenic Engineering Conference Board was both generous and skillful in helping to initiate this new conference and their guidance and acceptance is gratefully acknowledged. The Cryogenic Engineering Conference program chairman, M. J. Hiza, greatly facilitated the interaction for the two conferences and provided valuable assistance in generating a workable program. The proceedings of the 1975 Cryogenic Engineering Conference are published as Volume 21 of the *Advances in Cryogenic Engineering* and include many papers indicating innovative use of new cryogenic materials properties data.

The assistance of the many dedicated workers in the cryogenic materials field who have contributed to the reviewing of the final manuscripts for this volume is gratefully acknowledged by the editors. The list of all those individuals who have assisted in the many important tasks of readying the manuscripts for publication is as long as the list of conference attendees and any attempt to acknowledge individual contributions in this limited space would not do justice to their contributions.

From this collection of papers, it is easy to assess that two principal research directions are currently contributing stimuli for cryogenic materials research: applied superconductivity and the storage and transportation of LNG. Particularly the applied superconductivity programs, including power transmission, superconducting power packages, and fusion reactors, are heavily supported by federal agency interests and funding. Consideration of these applications requires that components operate reliably for extended periods of time at low temperatures. Future materials properties data will result from this requirement, providing stimulus for research programs related to efficient alloy selection, alternate materials choices, and degradation of properties, all leading to safer design.

The conception of an international conference devoted to materials research at low temperatures began within a national materials research program directed by the Cryogenics Division of the National Bureau of Standards. This program, "Materials Research in Support of Superconducting Machinery," is sponsored by the Advanced Research Projects Agency of the United States Department of Defense, under the direction of E. C. van Reuth, who, by his unflagging support and encouragement to ensure both excellence and relevance to cryogenic materials research, contributed immeasurably to the initiation of an international materials conference. Additional impetus was added by our friends in Europe, Japan, and the USSR, who have encouraged us to proceed with the organization of ICMC.

The nuclei of the First International Cryogenic Materials Conference were the researchers and designers who had previously participated in workshops held for the superconducting machinery materials program. However, it was the enthusiastic response of the world-wide materials research community with excellent presentations and papers that guaranteed a stimulating conference and useful proceedings. The conference contained many exchanges of ideas to provide insight and direction for future low-temperature materials research.

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A RESEARCH PROGRAM ON THE PROPERTIES OF STRUCTURAL MATERIALS AT 4 K*

R. P. Reed and A. F. Clark

Cryogenics Division
NBS Institute for Basic Standards
Boulder, Colorado

and

E. C. van Reuth

Advanced Research Projects Agency
Arlington, Virginia

For several years the United States has had experimental programs in superconducting machinery to produce both ac and dc generators and motors. One limitation to the progress of these efforts has been the lack of mechanical and thermal properties data for materials at liquid helium temperature. For the first time, machinery was to be built that must reliably operate for periods of 20 to 30 years at temperatures near 4 K. The complete absence of fracture data and scarce thermal property information seriously limited efficient design, and material selection was confined to a few alloys familiar to designers. From these considerations, a new program was developed by the Cryogenics Division of the National Bureau of Standards with the encouragement and support of the Advanced Research Projects Agency of the Department of Defense. This program to characterize structural materials' thermal and mechanical behavior from 4 to 300 K has now been in progress for one-and-a-half years, and is expected to continue for a similar period.

The program has three principal objectives: (1) to evaluate candidate structural materials for use in superconducting electrical machinery by measuring their mechanical and physical properties between 4 and 300 K and determining the effects on these properties of processing and joining; (2) to explore new materials, such as composites, for potential innovative design applications by performing screening tests on their low-temperature properties; and (3) to assist the information transfer of the available low-temperature properties data into design use by compiling and publishing those literature data that are available and by assessing what properties need further study.

The research on mechanical properties is directed toward adequate characterization of structural material behavior at 4 K. Naturally, the common tensile and fatigue properties of candidate materials are measured. In many cases, e.g., torque

* Invited paper.

tubes of superconducting rotating machinery [1], an essential material design factor is the elastic modulus. The complete set of elastic constants (Young's, shear, and bulk moduli, and Poisson's ratio) are being measured from 4 to 300 K for structural alloys and composites. Acoustic resonance and pulse–echo techniques have been adapted to determine rapidly and accurately these elastic moduli for alloys, semicontinuously from 4 to 300 K [2]. Previously many time-consuming tensile tests were necessary to measure the elastic moduli. Figure 1 shows typical elastic constant behavior at low temperatures, in this case for an annealed 5083 aluminum alloy [3]. Typically, the elastic constants of structural alloys have a linear dependence on temperatures near ambient, approach absolute zero with zero slope, and increase in magnitude approximately 10% from ambient temperature to 4 K. Magnetic transitions or crystalline phase transformations add complexity and anomalies to this normal behavior.

An important area of materials properties research, particularly for long-term reliability, is fracture. Fracture mechanics test techniques are employed to obtain fatigue crack growth rate and fracture toughness data on principal structural alloys; this represents the first such data obtained at 4 K [4–8]. To obtain usable valid data on K_{IC}, the critical plane strain fracture, toughness parameter, extremely thick (25 to 100 cm) specimens are needed to ensure plane strain fracture conditions in cryogenic alloys. Such large-specimen testing is extremely expensive and impractical. It was necessary, therefore, to incorporate the very recently developed *J*-integral test procedure to obtain useful fracture toughness data for the tough cryogenic alloys. Using *J*-integral techniques, designed to measure the critical amount of energy needed to extend a crack concomitant with plastic deformation, calculations of K_{IC} could then be obtained. Typical fracture toughness data [4] for three types of structural alloys, each having a different crystal structure, are illustrated in Fig. 2. Here, all data except for the titanium alloy were obtained from *J*-integral fracture test techniques. Notice that the face-centered cubic (fcc) structures are the toughest

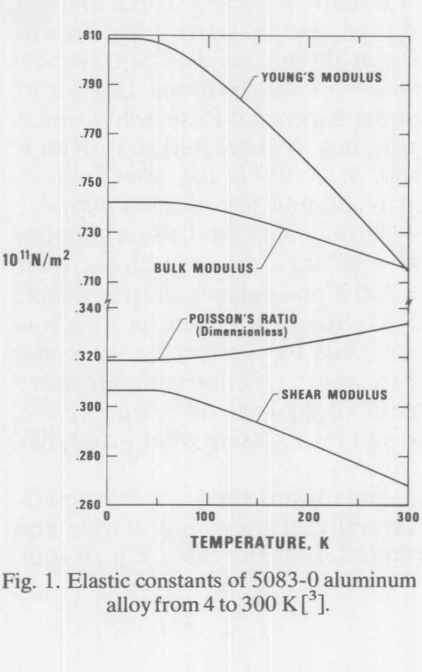

Fig. 1. Elastic constants of 5083-0 aluminum alloy from 4 to 300 K [3].

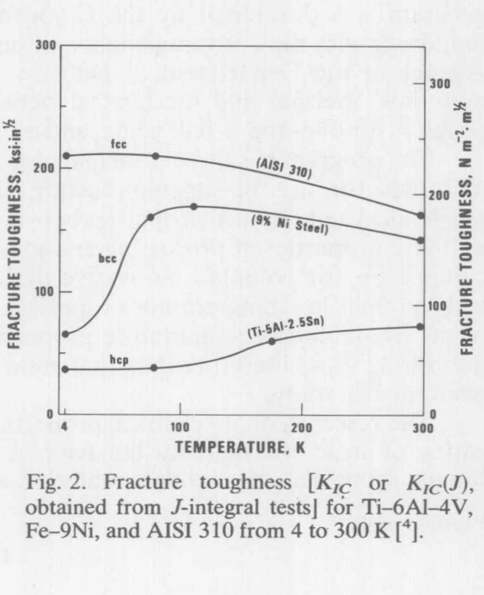

Fig. 2. Fracture toughness [K_{IC} or $K_{IC}(J)$, obtained from *J*-integral tests] for Ti–6Al–4V, Fe–9Ni, and AISI 310 from 4 to 300 K [4].

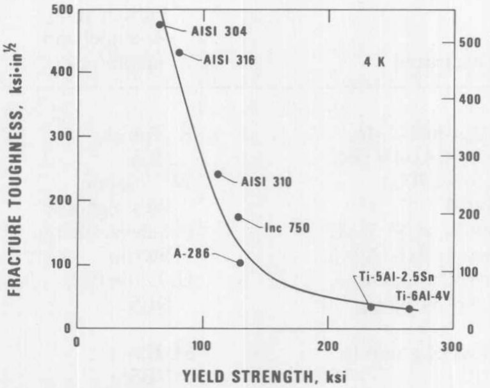

Fig. 3. Relation between fracture toughness (obtained from *J*-integral tests) and yield strength, both measured at 4 K, for some structural alloys tested in this program [4–8].

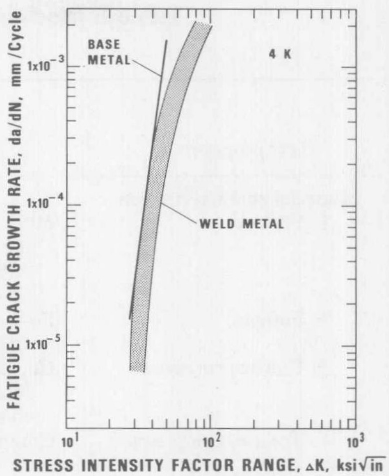

Fig. 4. Fatigue crack growth rate behavior of Inconel 750 base metal and electron beam and gas tungsten arc weld metal at 4 K [7].

material class at cryogenic temperatures. Also, fracture toughness correlates with yield strength (Fig. 3); higher yield strength alloys are less tough. The structural designer must have well-characterized materials in order to achieve the proper balance between strength and reliability.

Another neglected area of research at low temperatures has been that of weld properties. In many applications it is desirable to use welded structures, but knowledge of weld fracture characteristics at 4 K has been nonexistent. In some cases this has led to virtual exclusion of weld joints in prototype machines. Therefore, for selected alloys, fracture toughness and fatigue crack growth rates are being measured on weld configurations [8]. In Fig. 4, fatigue crack growth rate data at 4 K are compared for base metal and weld metal (electron beam and gas tungsten arc) of an Fe–Ni–Cr age-hardenable superalloy. Notice that in all stress ranges the fatigue cracks propagate faster at 4 K in the base metal than in the weld metal.

A complete outline of the mechanical properties, the alloys being measured, and the associated laboratories is shown in Table I.

There are many thermophysical properties that are essential for proper superconducting machine design. In thermal stress calculations for cooldown and quenching, it is necessary to know the specific heat, thermal conductivity, and thermal expansion of the component materials. Figure 5 compares the thermal expansion of selected alloys, several of which were measured in different conditions to determine the effects of processing. The effects of processing can be several percent (5–15%) [9] and can be readily accounted for, and even predicted, from room temperature data. Compared with other thermal properties, the characterization of thermal contraction of most structural alloys at low temperatures has less variation. Using Fig. 5, the difference between a low-expansion titanium alloy and a high-expansion copper (or aluminum) alloy is about a factor of two.

For transient thermal behavior analysis, thermal diffusivity [thermal conductivity/(specific heat × density)] data within the temperature range 4–300 K are needed. In Fig. 6, selected materials having a wide range of thermal conductivity

Table I. Mechanical Properties Test Programs for Fiscal Year 1975

Test program	Materials measured	Participating personnel and laboratories
Material characterization		
1. Fracture	5083 Al, Fe–21Cr–6Ni–9Mn, Inconel 718, Ti–6Al–4V (B), AISI 304, Inconel 706, Cu–0.4Cr–0.4Cd	R. Tobler, NBS W. Logsdon, Westinghouse
2. Fatigue	Fe–21Cr–6Ni–9Mn, AISI 304L, AISI 310, Inconel 718, A286	F. Schwartzberg, Martin
3. Elastic properties	Cu, Cu–Ni alloys, Cu–Sn alloys, Inconel 718, Nb–Ti alloys, composites	H. Ledbetter, NBS
4. Tensile, compression	Composites, alloys of group Ia	M. Kasen, NBS W. Logsdon, Westinghouse
Metallurgical variables		
1. Effect of grain size	Inconel 718	J. Wells, Westinghouse
2. Effect of cold work	Kromarc 58	J. Wells, Westinghouse
Mechanical variables		
1. Fatigue parameters	Fe–21Cr–6Ni–9Mn, Inconel 718, AISI 310	R. Tobler, NBS
2. Effect of thickness	2219 Al	D. Read NBS
Fabricated joints		
1. Welds	Inconel 718, 706, and 750	J. Wells, Westinghouse
2. Adhesive joints	Composites to aluminum	W. Hillig, General Electric

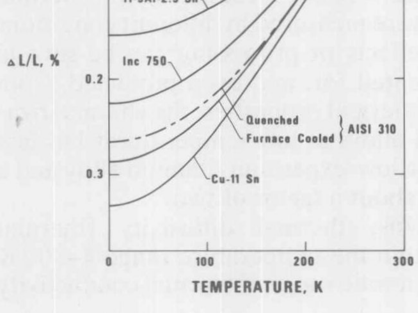

Fig. 5. Low-temperature thermal expansion of selected alloys.

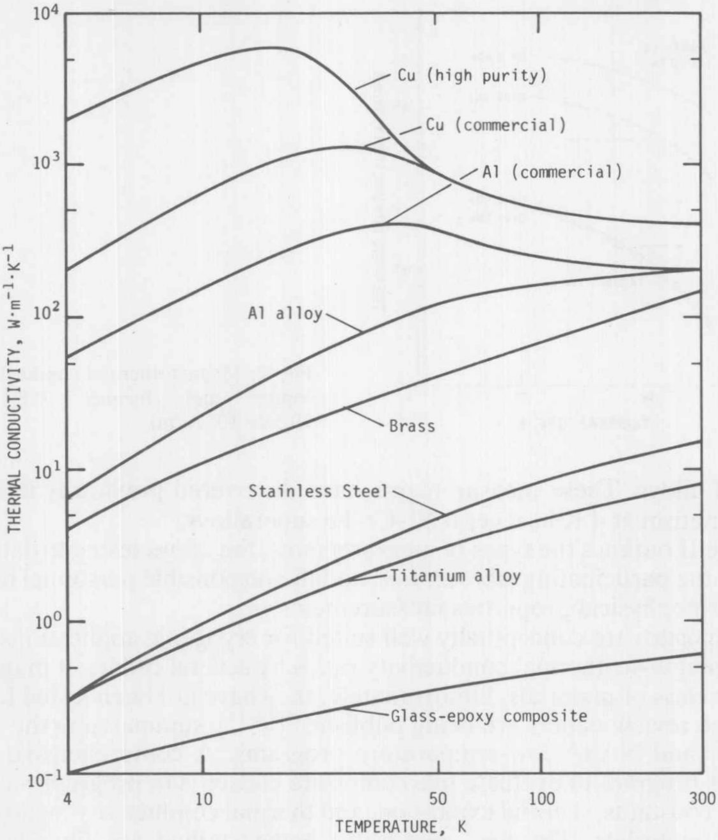

Fig. 6. Low-temperature thermal conductivity of selected materials.

at low temperatures are presented. The thermal conductivity of alloys is usually not sensitive to thermal treatment, varying by about 10% [10]. As the temperature is decreased, the alloy thermal conductivity decreases approximately linearly. The temperature dependence of purer elements is complex; the thermal conductivity initially increases as the temperature is decreased, reaches a maximum, then rapidly decreases as the temperature approaches absolute zero (e.g., copper in Fig. 6). Specific heat data [9] show less variation with composition and prediction of low-temperature behavior is possible to within 5 to 15%. At low temperature, the specific heat has a T^3 temperature dependence, which is reduced to a linear dependence as the temperature approaches room temperature.

Many components are exposed to magnetic fields; therefore it is essential to know the effect of magnetic fields on the thermal conductivity. Since no magnetothermal conductivity measurements have ever been made on alloys, a program was included to obtain such data [11]. Figure 7 shows some of these new data and that, indeed, the effect of an imposed magnetic field can result in a significant reduction (about 50%) for high-conductivity copper and should be considered in design. Finally, because designs must include magnetic field-shape and loss predictions, the magnetic susceptibility and the electrical resistivity are being measured for the