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Materials for Cold Neutron Sources: Cryogenic and Irradiation Effects

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ABSTRACT*

Materials for the construction of cold neutron sources must satisfy a range of demands. The cryogenic temperature and irradiation create a severe environment. Candidate materials are identified and existing cold sources are briefly surveyed to determine which materials may be used. Aluminum- and magnesium-based alloys are the preferred materials. Existing data for the effects of cryogenic temperature and near-ambient irradiation on the mechanical properties of these alloys are briefly reviewed, and the very limited information on the effects of cryogenic irradiation are outlined. Generating mechanical property data under cold source operating conditions is a daunting prospect. It is clear that the cold source material will be degraded by neutron irradiation, and so the cold source must be designed as a brittle vessel. The continued effective operation of many different cold sources at a number of reactors makes it clear that this can be accomplished.

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I. INTRODUCTION

A cold neutron source presents a unique set of demands for the material of construction. In addition to the extremely low temperatures at which the cold source operates, an intense flux of neutrons will be present. The combination of these factors results in a very severe environment. This paper provides a survey of the materials that might be used in construction of a cold source, and outlines some of the problems that may arise. In addition, existing cold sources are briefly surveyed, and the materials used in these cold sources are described. Finally, the most promising candidate materials are identified, and some potential problems are mentioned.

II. MATERIAL REQUIREMENTS

The requirements for a structural material for a cold neutron source are severe. First and foremost, the material must allow the passage of neutrons into and out of the cold source. Any absorption of neutrons will degrade the efficiency of the device. The material must withstand the stresses imposed, which will vary depending on the design of the cold source. This problem is exacerbated by the very low temperatures at which the cold source operates, and the thermal cycles which may occur during long term operation. In order to minimize the demands on the refrigeration system the cold source material should have a low heat generation rate due to irradiation and a high thermal conductivity. The material must be compatible with the cold source moderator. The material should be readily available, and preferably relatively inexpensive. Construction of the cold source vessel demands that the material be readily fabricated, which may entail welding of relatively thin sheets. Forming and welding must not have adverse effects on the mechanical properties. The resultant structure must be leak- and vacuum-tight. Finally, other safety issues such as flammability and accident scenarios must be addressed. The result is a demanding set of design constraints.

The paramount importance of low neutron absorption eliminates several classes of alloys, including iron-, copper-, nickel-, and titanium-based alloys. Zirconium-based alloys suffer from low thermal conductivity, making them unattractive. As a result of this process of elimination, aluminum- and magnesium-based alloys are the apparent candidate materials.

III. EXISTING COLD SOURCES

A brief survey of existing cold sources(1-5) is given in Table I. For comparison, the planned Advanced Neutron Source (ANS) Reactor at Oak Ridge will operate at 350 MW and the cold source will be exposed to a thermal flux of 4×10^{19} neutrons/($m^2 \cdot s$). In Table II the materials used for these cold sources and additional comments are listed(1-9). These tables indicate that aluminum alloys are the most common choice. Both 5000- and 6000-series alloys have been used successfully. Magnesium alloys have been used also.

TABLE I. EXISTING COLD SOURCES

Reactor	Reactor Power (MW)	Thermal Flux [$n/(m^2 \cdot s)$]	Moderator			
			Type	Thickness (mm)	Volume (L)	Heat removed (W)
HFR Grenoble	57	6×10^{18}	LD_2	380	25	5000
HFBR Brookhaven	60	3×10^{18}	LH_2 subcooled	66	1.35	600
Orphee Saclay	14	3×10^{18}	LH_2	50		500
FRJ2 Julich	15	9×10^{17}	$LH_2 + LD_2$	55	0.85	1000
DR3 Riso	10	7×10^{17}	H_2 supercritical	60	0.57	620
FR2 Karlsruhe	43	5×10^{17}	LH_2	45	0.25	50
EL3 Saclay	17	5×10^{17}	LH_2	70	0.40	80
DIDO Harwell	15	4×10^{17}	LH_2	30	0.20	40
Herald Aldermaston	5	1×10^{17}	$LH_2 + LD_2$	190	3	200
RRI Kyoto			LH_2	154	4	70
NBSR NIST			D_2O/H_2O ice	330	30	

TABLE II. MATERIALS USED IN EXISTING COLD SOURCES

Reactor	Material	Comments
HFR Grenoble	Pure aluminum Zircaloy 2	Commercial purity; inner vessel Outer containment
HFBR Brookhaven	6061-T4 Al	Heat treated after brazing assembly
Orphee Saclay	A286	Fe-26Ni-15Cr stainless steel
FRJ-2 Julich	5052 Al	Electron beam welded, also used for vacuum jacket
DR3 Riso	5052-O Al	Limit 4.5 × 10^{26} n/m^2, ≈20 years Outer containment also
EL3 Saclay	5052 Al	
DIDO Harwell	Magnesium	
Herald	5056 Al	Also for vacuum chamber
RRI Kyoto	5052 Al	
NBSR NIST	AZ31B Mg	Limited by existing refrigeration

IV. ALUMINUM ALLOYS

Material Description and Cryogenic Properties

There are several classes of aluminum alloys to consider. However, only the 5000- and 6000-series alloys are feasible candidates. The 2000-series (Al-Cu) has low toughness at cryogenic temperatures, the 3000-series (Al-Mn) has low strength, and the 7000-series (Al-Zn-Mg-Cu) has poor ductility and toughness at cryogenic temperatures. The 5000-series (Al-Mg) is used extensively for cryogenic applications. These non-heat-treatable alloys are readily weldable. They have relatively low strength but high toughness over all cryogenic temperatures. The 5052 alloy contains 2.5 wt % Mg and 0.25 wt % Cr. Other possibilities include 5154 (3.5Mg-0.25Cr), 5456 (5.1Mg-0.12Cr-0.8Mn) and 5083 (4.4Mg-0.15Cr-0.7Mn). The 6000-series alloys (Al-Mg-Si) are also widely used in cryogenic applications. These medium-strength alloys are heat-treatable with strengthening provided by the precipitation of Mg_2Si particles in the matrix, and can be welded, although a postweld heat treatment is necessary if the weld joint is to match the strength of the precipitation-hardened base metal. Possible alloys include 6061 (1.0Mg-0.6Si-0.28Cu-0.20Cr) and 6063 (0.7Mg-0.4Si).

The cryogenic properties of these alloys are well-documented, particularly for 6061. Data are available from a number of handbooks and other sources(10-13). The tensile properties(14-16) of 6061-T6, the peak strength condition for this alloy, are shown in Fig. 1. The properties of 5456-H321 are shown in Fig. 2 for comparison(16,17). The yield strength and ductility of these alloys tends to increase as the temperature is lowered. The high values of the notched-tensile

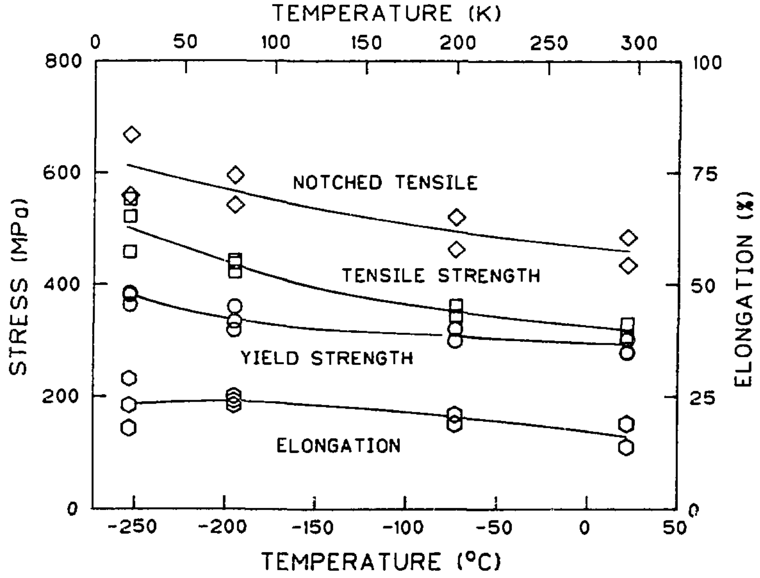

Fig. 1. Tensile properties vs temperature for 6061-T6 aluminum. Stress concentration factor for notched tensile specimens is K_t = 8.0 or 6.3. ***Sources***: W. Weleff, H. S. McQueen, and W. F. Emmons, "Cryogenic Tensile Properties of Selected Aerospace Materials," pp. 14-25, and J. L. Christian and J. F. Watson, "Mechanical Properties of Several 2000 and 6000 Series Aluminum Alloys," pp. 63-76 in *Advances in Cryogenic Engineering*, Vol. 10, Proceedings of the 1964 Cryogenic Engineering Conference, K. Timmerhaus, Ed., Plenum, New York, 1965; and M. P. Hanson, G. W. Stickley, and H. T. Richards, "Sharp-Notch Behavior of Some High-Strength Sheet Aluminum Alloys and Welded Joints at 75, -320, and 423°F," p. 3 in *Symposium on Low-Temperature Properties of High-Strength Aircraft and Missile Materials, ASTM STP 287*, American Society for Testing and Materials, Philadelphia, 1961.

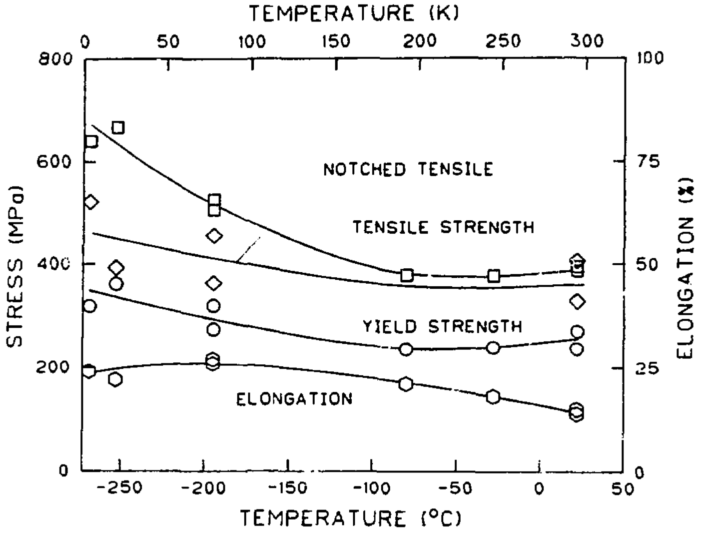

Fig. 2. Tensile properties vs temperature for 5456-H321 aluminum. Stress concentration factor for notched tensile specimens is K_t = 16. ***Sources***: M. P. Hanson, G. W. Stickley, and H. T. Richards, "Sharp-Notch Behavior of Some High-Strength Sheet Aluminum Alloys and Welded Joints at 75, -320, and 423°F," p. 3 in *Symposium on Low-Temperature Properties of High-Strength Aircraft and Missile Materials, ASTM STP 287*, American Society for Testing and Materials, Philadelphia, 1961; and J. G. Kaufman, K. O. Bogardus, and E. T. Wanderer, "Tensile Properties and Notch Toughness of Aluminum Alloys at -452°F in Liquid Helium," p. 294 in *Advances in Cryogenic Engineering*, Vol. 13, Proceedings of the 1967 Cryogenic Engineering Conference, K. D. Timmerhaus, Ed., Plenum, New York, 1968.

data indicate that these alloys are not notch-sensitive, which suggests that they will have good toughness. A comparison of the ratio of the notched tensile strength to the yield strength as a function of the yield strength at 4 K for a number of aluminum alloys is shown in Fig. 3. The 5000- and 6000-series alloys(18) are used for cryogenic applications because of their high notch-yield ratios.

The toughness of these alloys has been examined with notched tensile, tearing, and plane strain fracture toughness testing. The energy required for tearing of thin sheet specimens of 6061-T6 and 5456-H321 is shown(19) in Fig. 4. These data indicate that the toughness of the 6061 material actually increases as the temperature is reduced, and the toughness of the 5456 material is relatively constant. It should be noted that the fracture toughness of 6061-T6 (Fig. 5) is much lower than one might expect from the notched tensile and tear test data(20,21).

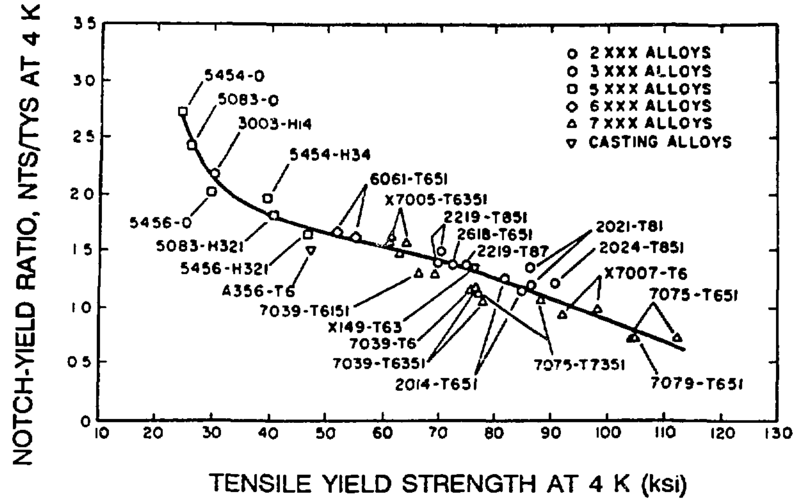

Fig. 3. Notch-yield ratio vs tensile yield strength for aluminum alloys at 4 K. Stress concentration factor for notched tensile specimens is $K_t = 16$. *Source*: J. G. Kaufman and E. T. Wanderer, "Tensile Properties and Notch Toughness of Some 7XXX Alloys at -452°F," p. 27 in *Advances in Cryogenic Engineering*, Vol. 16, Proceedings of the 1970 Cryogenic Engineering Conference, K. D. Timmerhaus, Ed., Plenum, New York, 1971.

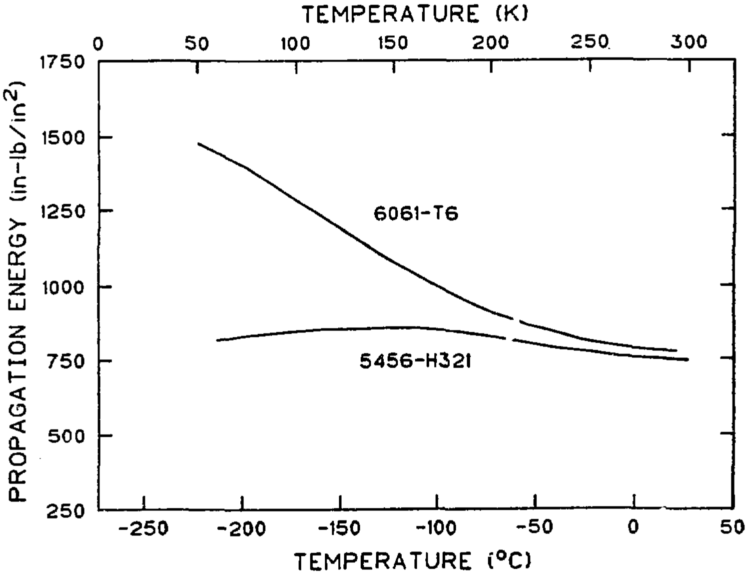

Fig. 4. Tear resistance of 6061-T6 and 5456-H321 at low temperatures. *Source*: J. G. Kaufman and M. Holt, "Evaluation of Fracture Characteristics of Aluminum Alloys at Cryogenic Temperatures," pp. 77-85 in *Advances in Cryogenic Engineering*, Vol. 10, Proceedings of the 1964 Cryogenic Engineering Conference, K. Timmerhaus, Ed., Plenum, New York, 1965.

However, these fracture toughness data are for thick sections only, and material in a thin-walled cold source should have a higher toughness, since fracture would occur under plane-stress conditions.

These aluminum alloys are readily weldable under appropriate conditions. The mechanical properties of welds have been studied at cryogenic temperatures by several investigators(10,11,16,22-24).

Effects of Radiation

The effects of irradiation on these alloys have been studied to some extent. Limited data are available for irradiations slightly above room temperature on 6061, 6063, 5052 and 5154 alloys(25-32). These data indicate that the yield and ultimate strengths are increased by

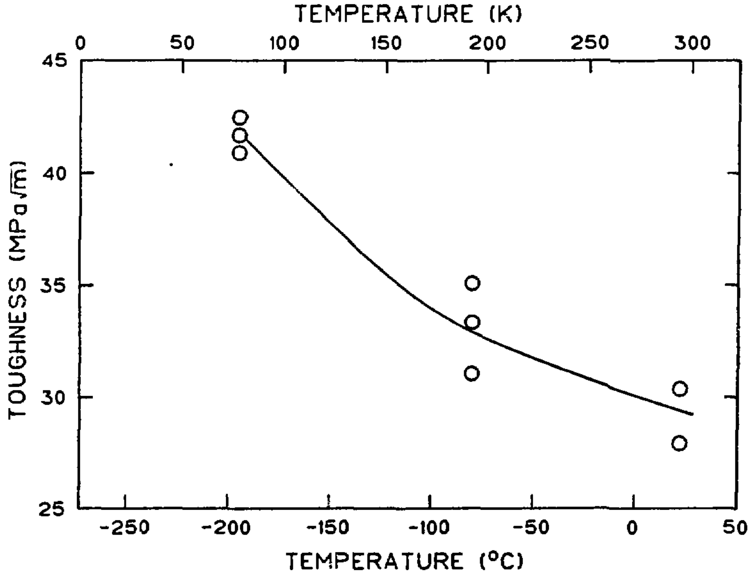

Fig. 5. Plane-strain fracture toughness of 6061-T6 at low temperatures. *Source*: F. G. Nelson and J. G. Kaufman, "Plane Strain Fracture Toughness of Aluminum Alloys at Room and Subzero Temperatures," pp. 27-39 in *Fracture Toughness Testing at Cryogenic Temperatures, ASTM STP 496*, American Society for Testing and Materials, Philadelphia, 1971.

irradiation whereas the ductility is greatly reduced. Data(27,31) for 6061-T6 and 5052-0 are shown in Figs. 6 and 7, respectively. The primary mechanism for these changes is the production of silicon transmuted from the aluminum matrix by thermal neutrons. In unalloyed aluminum and in 6061-T6, the silicon forms tiny particles of elemental silicon in the matrix, resulting in increased strengths. In the 5000-series alloys the silicon reacts with magnesium in solution to form precipitates of Mg_2Si. Thus, the 5000-series alloys may be more greatly affected than quenched-and-tempered 6000-series alloys, which do not have excess magnesium present in the matrix. Since thermal neutrons are responsible for the silicon generation, the thermal neutron flux may play a greater role in embrittlement than the fast neutron flux(25,28,31), at least at temperatures where migration of silicon can occur. Generation of point defects by the fast neutrons will increase the mobility of the silicon. Above room temperature, point defects are mobile in aluminum, and the majority are annihilated by recombination. However, these diffusional processes will be severely curtailed at the low temperatures at which the cold source will operate. Under such conditions, point defects and transmuted silicon atoms will be frozen-in, and the degree of hardening per unit flux may be much greater than at high temperatures. This is evident in the very limited cryogenic irradiations of aluminum alloys that have been carried out(33-36) to a maximum fast fluence of only 10^{21} neutrons/m^2 at 16 K for 6061-T6 and 5456-H321. The yield strength at 16 K of the 6061 alloy increased from 330 MPa unirradiated to 390 MPa after irradiation whereas the 5456 alloy showed a much greater increase (from 300 to 460 MPa). Irradiations to these low fluences at 60°C would cause no change in yield strength(27,31,32) (see Figs. 6 and 7). The ultimate tensile strengths for the 6061 and 5456 alloys were essentially unaffected (450 and 640 MPa, respectively), as was the total elongation to failure (28 and 16%, respectively). Limited tests at the Grenoble reactor(37) indicate that cryogenic irradiation results in severe embrittlement. No data exist that show the effect of irradiation by cold neutrons at either near-ambient or cryogenic temperatures.

The considerable experimental problems associated with cryogenic irradiations make such data very difficult to generate. Even warming the specimen to liquid nitrogen temperatures (77 K) will result in significant recovery, as pointed out in a review article(38). Thus, one is essentially

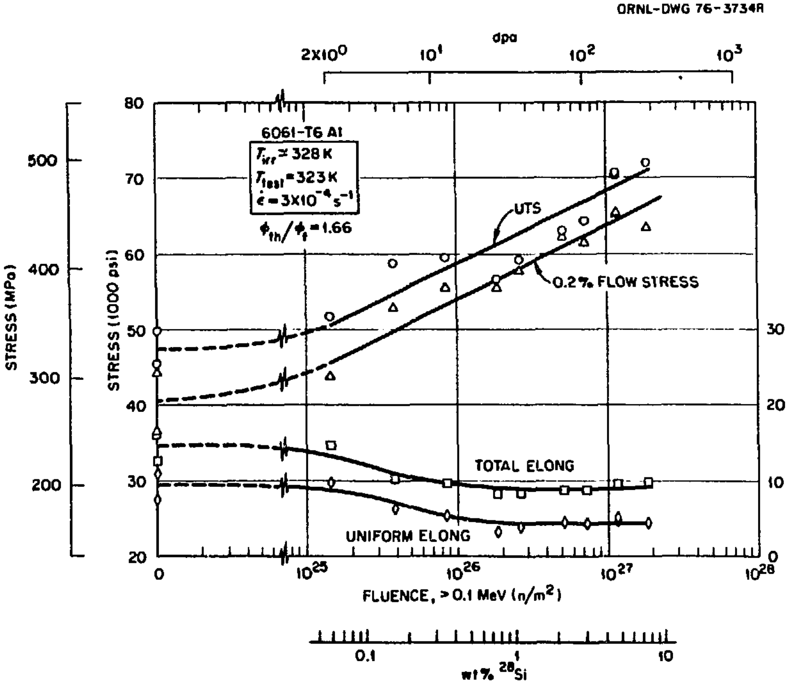

Fig. 6. Effect of fast fluence (>0.1 MeV) on tensile properties of 6061-T6 at 323 K. *Source*: K. Farrell and R. T. King, "Tensile Properties of Neutron-Irradiated 6061 Aluminum Alloy in Annealed and Precipitation-Hardened Conditions," pp. 440-49 in *Effects of Irradiation on Structural Materials, ASTM STP 683*, J. A. Sprague and D. Kramer, Eds., American Society for Testing and Materials, Philadelphia, 1979.

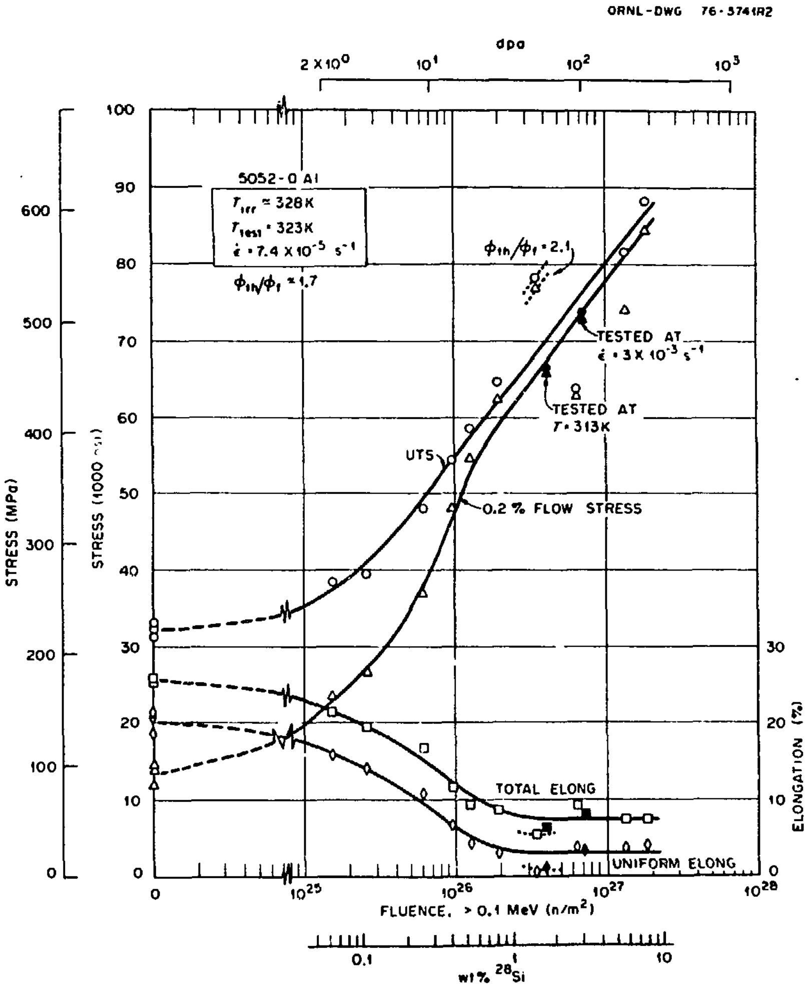

Fig. 7. Effect of fast fluence (>0.1 MeV) on tensile properties of 5052-O at 323 K. *Source*: K. Farrell, "Microstructure and Tensile Properties of Heavily Irradiated 5052-0 Aluminum Alloy," *Journal of Nuclear Materials* 97, 33-43 (1981).

forced to consider the cold source material as brittle, and design accordingly. Even with annealing treatments between cold cycles to remove the irradiation damage, the material will rapidly reembrittle.

V. MAGNESIUM ALLOYS

The magnesium alloys can be divided into two groups, those which contain zirconium, added as a grain refining agent, and those which do not. Aluminum is often added to the latter group to increase the strength. A common medium-strength alloy is AZ31B (3Al-1Zn-0.2Mn). This alloy is available in a range of sheet, plate and extrusions.

The mechanical properties at cryogenic temperatures have been measured for a number of magnesium alloys, and can be found in handbooks(12,39). In general, the yield and ultimate tensile strengths increase as the temperature is lowered, while the ductility decreases(39) as shown for extruded AZ31B-F in Fig. 8. Limited data(40,41) are also available for welded material.

The effect of irradiation has been studied for only a few alloys. While there has been a fair amount of work done on the Magnox alloys used in the British nuclear industry(42), other alloys have not received the same attention. Sturcken(30) irradiated a series of simple magnesium-aluminum alloys, and observed increases in the yield and ultimate strengths and very large decreases in the ductility.

The only available data for cryogenic irradiations involve basic studies of the recovery processes which accompany annealing(43,44) and studies of the deformation of single crystals(45,46). These studies indicate that recovery of point defects will begin at very low temperatures, as is the case for the aluminum alloys. No data exist for the effect of irradiation by cold neutrons on the mechanical properties of magnesium alloys.

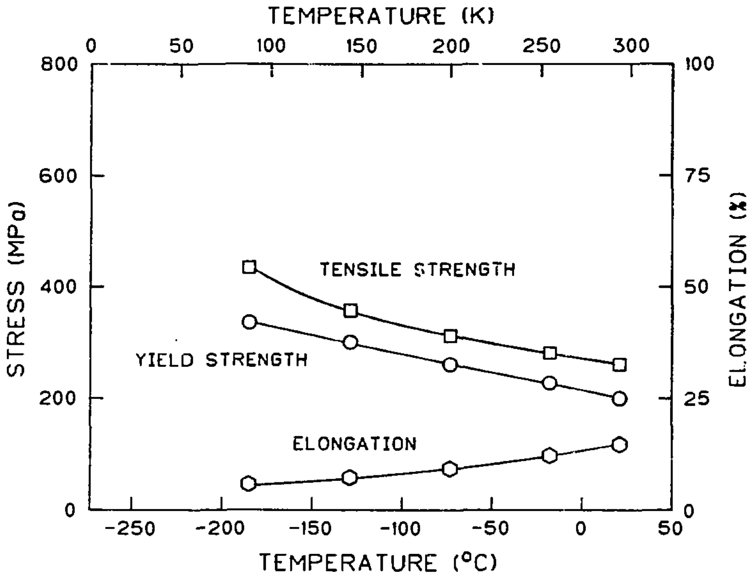

Fig. 8. Tensile properties vs temperature of AZ31B-F magnesium. *Source*: *Metals Handbook, Ninth Edition, Vol. 2, Properties and Selection: Nonferrous Alloys and Pure Metals*, p. 554, American Society for Metals, Metals Park, Ohio, 1979.

VI. DISCUSSION

There are very limited data for the mechanical properties of possible cold source structural materials at cryogenic (T < 25 K) irradiation conditions. All indications are that the material will be severely embrittled as a consequence of irradiation at low temperature. However, the fact that more than ten different cold sources have been successfully built and operated indicates that the problems of material selection and safe operation are not insurmountable. A common approach seems to be the provision of a secondary containment shield which will provide an additional layer of protection for the cold source vessel in addition to vacuum thermal insulation. Because of the degradation of the mechanical properties, the cold source vessel must be considered very brittle, and must be designed accordingly. It is apparent that this can be done successfully.

The generation of mechanical properties data for cryogenic irradiation conditions is a daunting task. The difficulties associated with maintaining the very low temperatures necessary to prevent annealing out the radiation damage during removal of the specimen from the irradiation facility are formidable. The common approach to the limited cryogenic irradiation mechanical property testing done in the past was to irradiate and test in situ using specially designed irradiation facilities. This limited the irradiation conditions to very low fluences and only relatively simple mechanical tests such as tensile or notched tensile tests could be conducted. Conducting a more complicated fracture mechanics test seems an unlikely possibility.

One avenue to consider is conducting irradiations at liquid nitrogen temperatures. It should be feasible to maintain this temperature during irradiation and subsequent transfer and testing. Although the mechanism of embrittlement at 77 K may be somewhat different than at much lower temperatures, this should be a step in the right direction. Even in this case, it may be difficult to find a facility for these irradiations.

Yield and ultimate tensile strengths can be measured with relatively simple apparatus at 77 K. Conducting a fracture mechanics test of thin sheet material will be much more difficult. The possibility of using a simple test such as a notched tensile or a tear test should be considered. This would require the development of a good correlation between the notched or tear test and the actual failure of a vessel, perhaps by conducting proof tests at 77 K by pressurizing intentionally flawed vessels. A dummy vessel could be built from an alloy with a silicon content equal to the estimated lifetime neutron-generated level of silicon. The vessel could be solution-treated to dissolve the silicon, and then quenched into liquid nitrogen to hold as much of the silicon in solution as possible, to simulate the very uniform distribution of silicon that would be produced by cryogenic irradiation. The vessel could then be tested to failure at 77 K. In any case, it seems prudent to design a cold source so that it can be removed from the reactor and replaced.

VII. SUMMARY AND CONCLUSIONS

Selection of a material for the construction of a cold neutron source requires satisfying many demanding criteria. Very little data exist for mechanical properties under irradiation conditions at low temperature. The favored material of construction appears to be either aluminum-magnesium or aluminum-magnesium-silicon alloys. These alloys offer adequate cryogenic mechanical properties in the unirradiated condition, and are readily formed and welded. The aluminum-magnesium-silicon alloys may be somewhat less sensitive to irradiation, but at cryogenic temperatures either alloy will quickly become embrittled. Generating mechanical property data for cryogenic irradiation conditions is a difficult task. Irradiation at 77 K may provide an indication of the effects, but will not fully duplicate the operating conditions. Materials under these conditions will rapidly embrittle, and thus it is necessary to consider the cold source as a brittle vessel. Despite these constraints, it is clear that it is possible to design and operate cold sources safely and effectively, as the operation of many different cold sources in many different reactors attests.

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