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SSD-TDR-63-371

Metals and Alloys for Cryogenic Applications - A Review

20 JANUARY 1964

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Materials Sciences Laboratory

Prepared for COMMANDER SPACE SYSTEMS DIVISION
UNITED STATES AIR FORCE
Inglewood, California

LABORATORIES DIVISION • AEROSPACE CORPORATION
CONTRACT NO. AF 04(695)-269

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This technical documentary report has been reviewed and is approved for publication and dissemination. The conclusions and findings contained herein do not necessarily represent an official Air Force position.

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ABSTRACT

An up-to-date review of metals and alloys suitable for cryogenic aerospace structural applications has been made. The mechanical properties of austenitic stainless steels, other steels, aluminum alloys, titanium alloys, nickel alloys and cobalt alloys from +78 to -423°F are presented, including tensile and yield strengths, elongation and notch/tensile ratios. Mechanical properties of weldments are also presented. The question of notch toughness and the notch acuity factor, K_t, is discussed with respect to low temperature tensile testing. Compatibility with the liquid gases is discussed and alloys most suitable for containing liquid oxygen and hydrogen in aerospace vehicles are recommended.

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I. INTRODUCTION

The aerospace materials engineer's interest in the properties of metals at cryogenic temperatures stems from the use of the liquid gases as propellants and pressurants in launch vehicles and spacecraft. These gases, which include helium, nitrogen, oxygen, fluorine, and hydrogen, are listed in the cryogenic temperature scale, Table 1, in order of decreasing boiling point.

Liquid nitrogen and helium are used as pressurants for fuel tanks and hydraulic activated systems. Liquid nitrogen is also used as a precooling agent to increase the gas storage capacity of the high-pressure helium vessels.

Table 2 is a listing of the fuels and oxidants in most of the missile and booster systems in being today. Liquid oxygen (LOX) is almost exclusively used in the liquid propellant systems except for the Titan II and III, which use N_2O_4. This preference for LOX is primarily due to its low cost. Liquid fluorine has been proposed as an advanced oxidant; systems using liquid fluorine are presently in the R and D stage. Liquid hydrogen, primarily because of its lower density than hydrazine and RP-1, is under consideration as a fuel for the upper booster stages.

The second stage of the Saturn I vehicle is made up of six Pratt and Whitney RL-10 engines (of the Centaur type) using liquid oxygen-liquid hydrogen ($LOX-LH_2$). The J-2 engine, used for the upper stages of Saturn 1-B and V, and the M-1 engine, designed for the upper stage of the NOVA vehicle, also use $LOX-LH_2$.

In addition to their use in meeting launch vehicle requirements, cryogenic liquids also appear promising, for spacecraft propulsion and system power applications.

Table 1. Cryogenic Temperature Scale

MATERIAL OR CONDITION	TEMPERATURE, DEGREES			
	F	R	C	K
ROOM TEMPERATURE	+78	538	+ 25	298.0
DRY ICE AND ALCOHOL	- 100	360	- 73	200.0
OXYGEN *	-297	163	- 183	90.0
FLUORINE *	-307	153	- 188	85.0
NITROGEN *	-320	140	- 195	77.0
HYDROGEN *	-423	37	-253	20.0
HELIUM *	-452	8	-269	4.2
ABSOLUTE ZERO	-460	0	-273	0.0

* TEMPERATURE IS BOILING POINT OF LIQUID GAS

Table 2. Propellants for Missiles and Boosters

SYSTEM	FUEL	OXIDIZER
ATLAS TITAN I THOR CENTAUR REDSTONE JUPITER NOVA SATURN	RP-1 AND/OR LIQUID HYDROGEN	LIQUID OXYGEN
TITAN II	HYDRAZINE-UMDH (AEROZINE 50)	N_2O_4
TITAN III	HYDRAZINE-UMDH (AEROZINE 50 + SOLIDS)	N_2O_4 + SOLIDS
POLARIS MINUTEMAN	SOLIDS	
NIKE-ZEUS PERSHING	SOLIDS	

With the requirements for storage and handling of these cryogenic liquids well established, investigators have over the past eight years conducted extensive tests in the search for storage and handling materials. The selection of these materials is based on many factors, including

a) strength-to-density ratio (room and low temperatures)
b) toughness (room and low temperatures)
c) weldability
d) formability
e) compatibility with the liquid
f) availability
g) cost

Prior to the development of the guided missiles, extremely little data were available on strength and toughness properties of engineering alloys at low temperatures. As a result of this paucity, much research has, during the past eight years, been directed toward determining these values. As a consequence, many books (Refs. 1-3), handbooks (Refs. 4 and 5), special publications (Refs. 6-9), literature and data reviews (Refs. 10-13), contract reports, scientific journal papers, and periodicals have appeared recently documenting this work. Primary among these, and worthy of special mention, is the Cryogenic Materials Data Handbook (Ref. 4). This handbook was initiated by the National Bureau of Standards Cryogenic Engineering Laboratory at Boulder, Colorado; responsibility has since been assumed by the Martin-Marietta Company, Denver, Colorado. This work emphasizes the graphical presentation of data and includes many thermophysical properties in addition to mechanical properties.

II. TESTING APPARATUS

After initiating programs to determine low-temperature mechanical properties, most laboratories found that cryostats, with which to perform the testing, were commercially unavailable. As a result, a variety of cryostats were individually built by the investigators; review of these units is reported by Schwartzberg in Ref. 14. Since the tension test can supply more useful information on elasticity, flow, fracture, ductility, and toughness with a single specimen than any other test, the majority of the units constructed have been tensile cryostats. Cryostats have ranged from the simple to the more sophisticated depending upon the needs of the individual laboratory. To obtain satisfactory thermal performance in the liquid H_2 and He range, it has been found necessary to make use of a combination of vacuum and liquid nitrogen insulation. A cryostat of this type, described by McClintock in Ref. 15, is shown in Figs. 1a and 1b.

The cryostat of Fig. 1 is a small unit constructed from a commercial stainless steel dewar. The major modification to the dewar is the incorporation of a tension linkage through the vacuum space at the bottom of the cryostat. A stainless steel bellows and universal joint are welded to the bottom of the inner can, permitting specimen contraction and alignment. Below the universal joint, a stack of 0.0005-in. thick washers is used to transmit the load from the lower grip to the specimen. A stud connected to the universal joint supports the stack from below, and a retaining well attached to the lower stem restrains the top of the stack. Therefore, the stack is compressed as tension is applied to a specimen within the cryostat. The increased heat path, caused by the numerous contact surfaces, substantially reduces the heat conduction from the stem through the vacuum jacket. Liquid consumption of this cryostat, after precooling with liquid nitrogen, is about 2.5 liters of hydrogen.

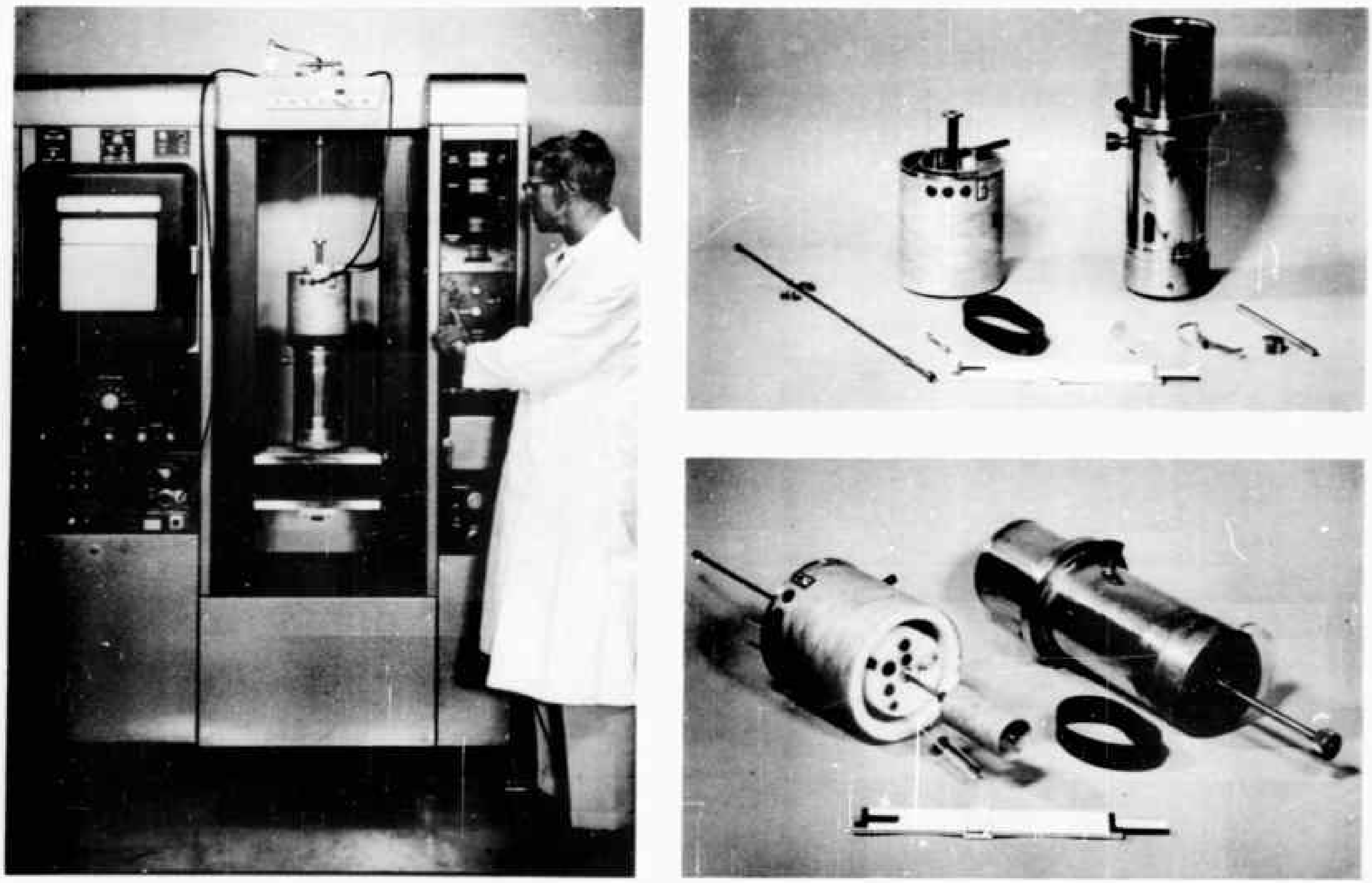

Fig. 1a. Tensile Cryostat

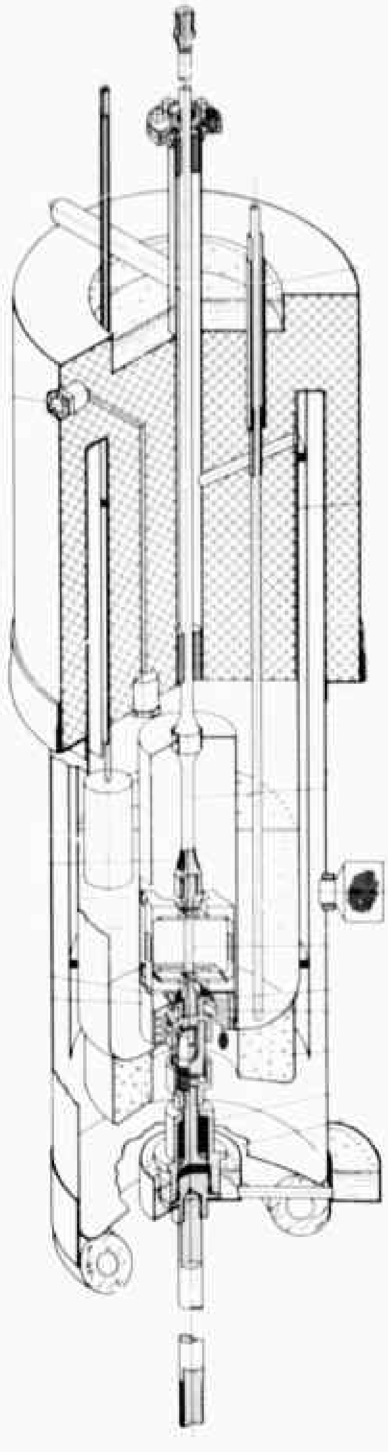

Fig. 1b. Cutaway of Tensile Cryostat

Other testing techniques, including fatigue, impact, torsion, shear, hardness, and biaxial, are reviewed by Schwartzberg in Ref. 16. Of these, the most important is the biaxial testing since it is well recognized that uniaxial mechanical property tests do not fully evaluate a material for structural service.

Until recently there was little need to measure strains at very low temperatures. As a result, information on the suitability of various strain gage materials for this application was lacking. In a recent work (Ref. 17), strain sensitivity changes, creep, drift, hysteresis effects, and data linearity were studied by mounting gates on constant-strain beams and loading them to strains up to ±2000 μ in./in. with a strain gage calibrator device. The more promising gage types were further tested to strains up to 13,000 μ in./in. on titanium tensile specimens. Tests were made for temperatures ranging from 78 to -423°F. Resistance changes were measured at temperatures down to liquid helium temperature (7°R).

The best gage type for the measurement of strains at a constant cryogenic temperature was a Nichrome V foil gage. This gage showed virtually no gage factor change from room to liquid hydrogen temperatures. Stabilized Armour D foil gages were found to be the best for the measurement of thermal strains in cryogenic environments because of the small resistance change. This gage showed a 1.5 percent increase in gage factor in compression and a 4 percent decrease in tension from room to liquid hydrogen temperature. Creep, drift, and hysteresis effects were small for both types of gages.

III. NOTCH TOUGHNESS

The notch tensile test is an important tool in understanding material toughness and brittle fracture. Consequently, to discuss the relative merits of engineering alloys for low-temperature applications, it is necessary to first describe the notch tensile test and its relationship to toughness and brittle fracture.

It has been well established that materials exhibiting ductility (high elongation and reduction in area) in a smooth bar tension test can fail catastrophically when subjected to impact or high localized stress loading at low temperatures. The ability of a material to resist brittle fracture is called toughness, and, as defined by the Modulus of Toughness, is a measure of the amount of work per unit volume required to carry a material to failure. Toughness can be represented by the total area under the complete stress-strain diagram and is expressed in units of in.-lb/in^3. Toughness can also be measured by the energy absorbed to rupture during an impact test. However, since all materials do not respond in the same way to variations in the speed of loading, different measures of toughness may be obtained from impact loadings than from static loadings.

In studying the ductile-to-brittle transition behavior of metals, it has been observed that the notch bar test gives a greater correlation between test and practice than any other test. This characteristic, coupled with the fact that no reliable impact test has yet been devised for thin sheet metal, has resulted in the notched tensile test being increasingly used to establish the necessary criterion for low-temperature materials evaluation.

The subject of fracture testing of high-strength sheet materials is one which is quite complex and still in the stages of research and development. Stimulated by the requirement for high-strength minimum weight cases for solid propellant motors, considerable research has been performed over the past five years in its behalf. In an attempt to establish testing standards for evaluating high-strength sheet materials with respect to their resistance to brittle fracture, the ASTM in 1959 formed a committee to review methods in use at that time.

The committee's review and recommendations were published in 1960 (Ref. 18). The reader is referred to this publication for a good overall discussion of the subject. Other works by leaders in the field include Irwin (Ref. 19), Neuber (Ref. 20), Peterson (Ref. 21), and more recently an analysis by Weiss and Sessler (Ref. 22).

The results obtained in a notched-tension test are extremely dependent on the test specimen geometry. The stress distribution in a notched specimen is characterized by the stress concentration factor, K_t, which defines the maximum stress at the notch root, σ_{max}, in terms of the notch stress or net section stress, σ_N, according to

$$\sigma_{max} = K_t \sigma_N$$ (See Fig. 2.)

and the stress gradient at the location of maximum stress

$$\left(\frac{d\sigma_y}{dx}\right)_{\sigma_y = \sigma_{max}} = -\frac{2\sigma_{max}}{r}$$

where r = root radius.

In recognizing this specimen geometry sensitivity, the Committee on Fracture Testing of the ASTM established standards for this testing and recently described a recommended notched tensile sheet specimen (Ref. 18), which is shown in Fig. 3; it contains a sharp notch (root radii of 0.001-in. maximum) with a resulting stress concentration factor of approximately 17. Unfortunately, for many years, investigators of low temperature properties have used less sharp notches, with K_t values as low as 2 and 3, and still today there is disagreement as to the nature of the notch and the K_t values to be used.

The chief argument against the use of very sharp notches is that practically all materials then can be made to appear to <u>lack</u> notch toughness. This lack

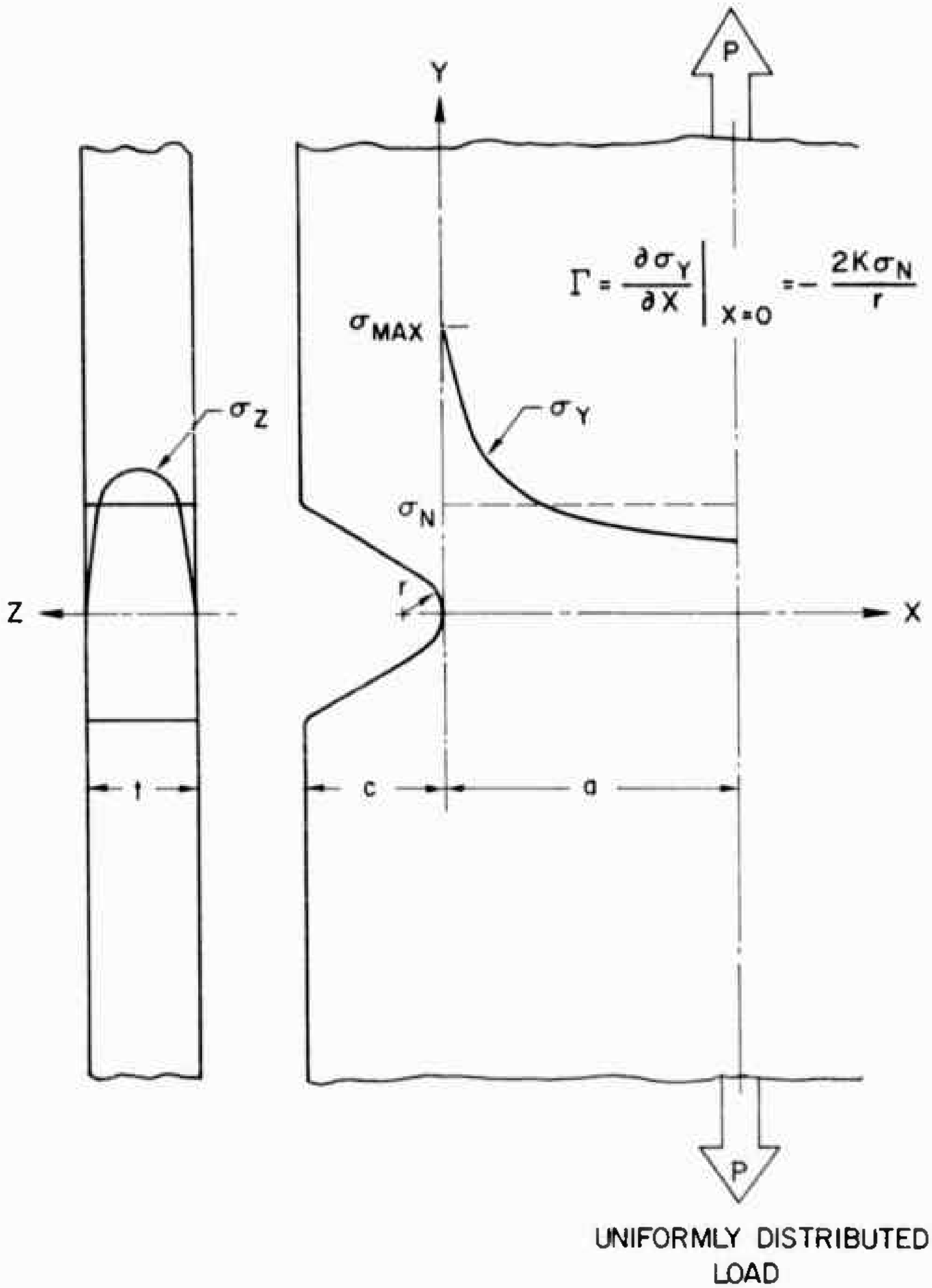

Fig. 2. Schematic Illustration of Stress Distribution for Notched Sheet Tension Specimen

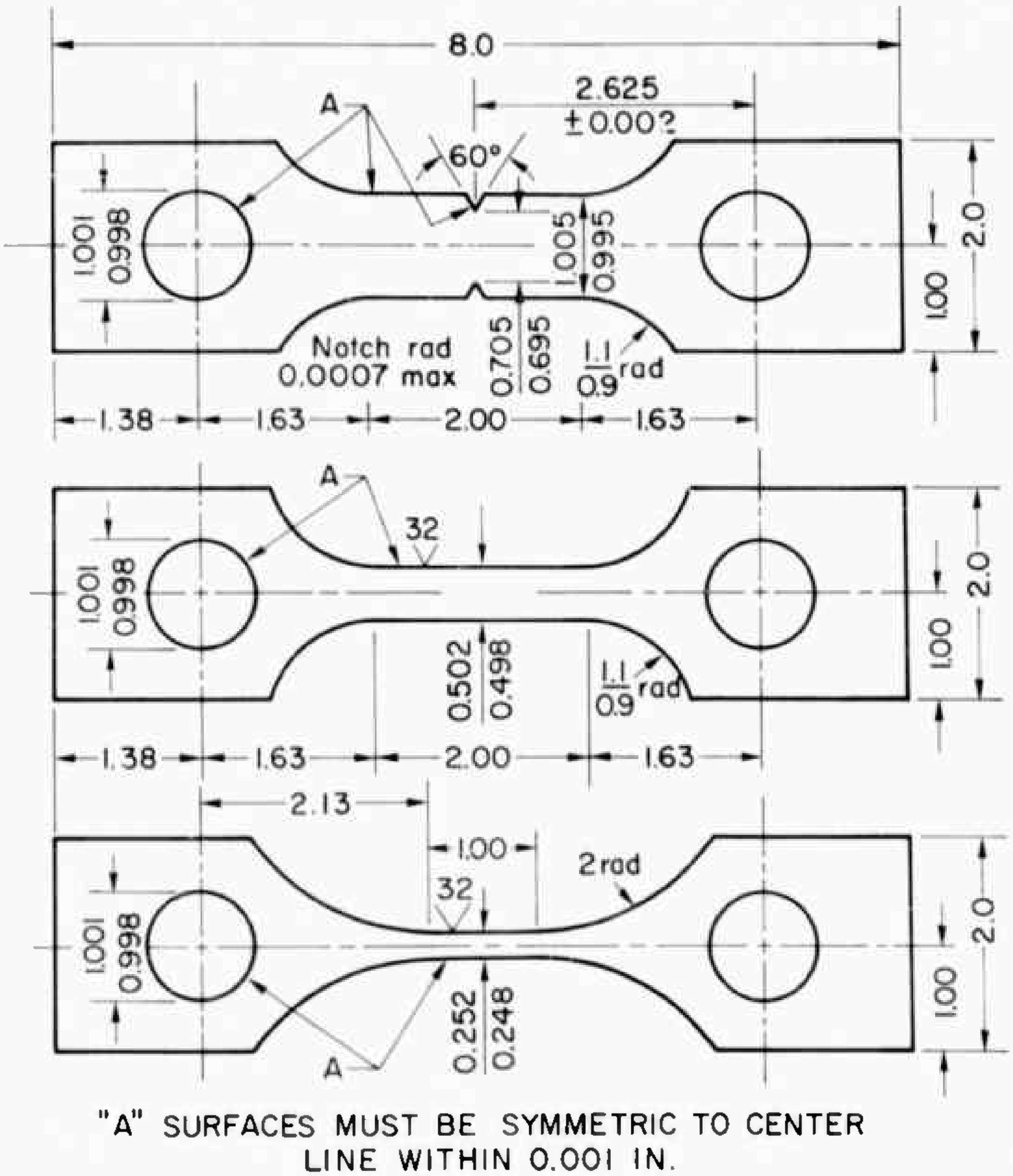

Fig. 3. Sharp-Edge-Notch and Smooth Tension Specimens

of agreement on the notch design to be used in the evaluation of materials has made it difficult to make cross comparisons of metals and alloys tested by different investigators on the basis of the notched strength-to-tensile strength (notch/tensile) ratio. Major proponents of the objection to the very sharp notch have published extensively (Refs. 23-26), and the basis of this stand has been summarized by Hurlich (Ref. 27). An attempt is made in Refs. 23-26 to correlate low-temperature notched tensile test data with that obtained on full-scale tank tests and also with the fatigue resistance of large welded joints. Tests were conducted with a K_t which they (Refs. 23-26) defined as

$$K_t = \left(\frac{a}{r}\right)^{1/2}$$

where

a = 1/2 distance between notches

r = radius at the root of notch

In tests conducted on stainless steels, the best correlation was obtained with a specimen having a K_t value of 6.3 (Fig. 4). The authors (Refs. 23-26) stated that this corresponds to values of 7.2 by the Peterson equation (Ref. 21) and 7.5 by the Neuber concept (Ref. 20). The data obtained are shown in Table 3, which presents results of fatigue tests on welded joint specimens tested at 78, -320, and -423°F and the results of notched-to-unnotched tension tests conducted on unwelded base metal coupons tested at these same temperatures. The three materials tested were 60% cold-rolled (CR) type 301 stainless steel sheet, modified by the addition of 0.15% nitrogen (both of these steels containing approximately 17% chromium and 7% nickel), and 75% CR type 310 stainless steel sheet (25% chromium and 20% nickel), all 0.020-in. thick. All fatigue tests were performed by cyclically loading the specimens from 0 to 140,000 psi at a rate of 6 cpm. This stress level is approximately 90 percent of the yield strength, and the test conditions are in the high stress - low cycle range.

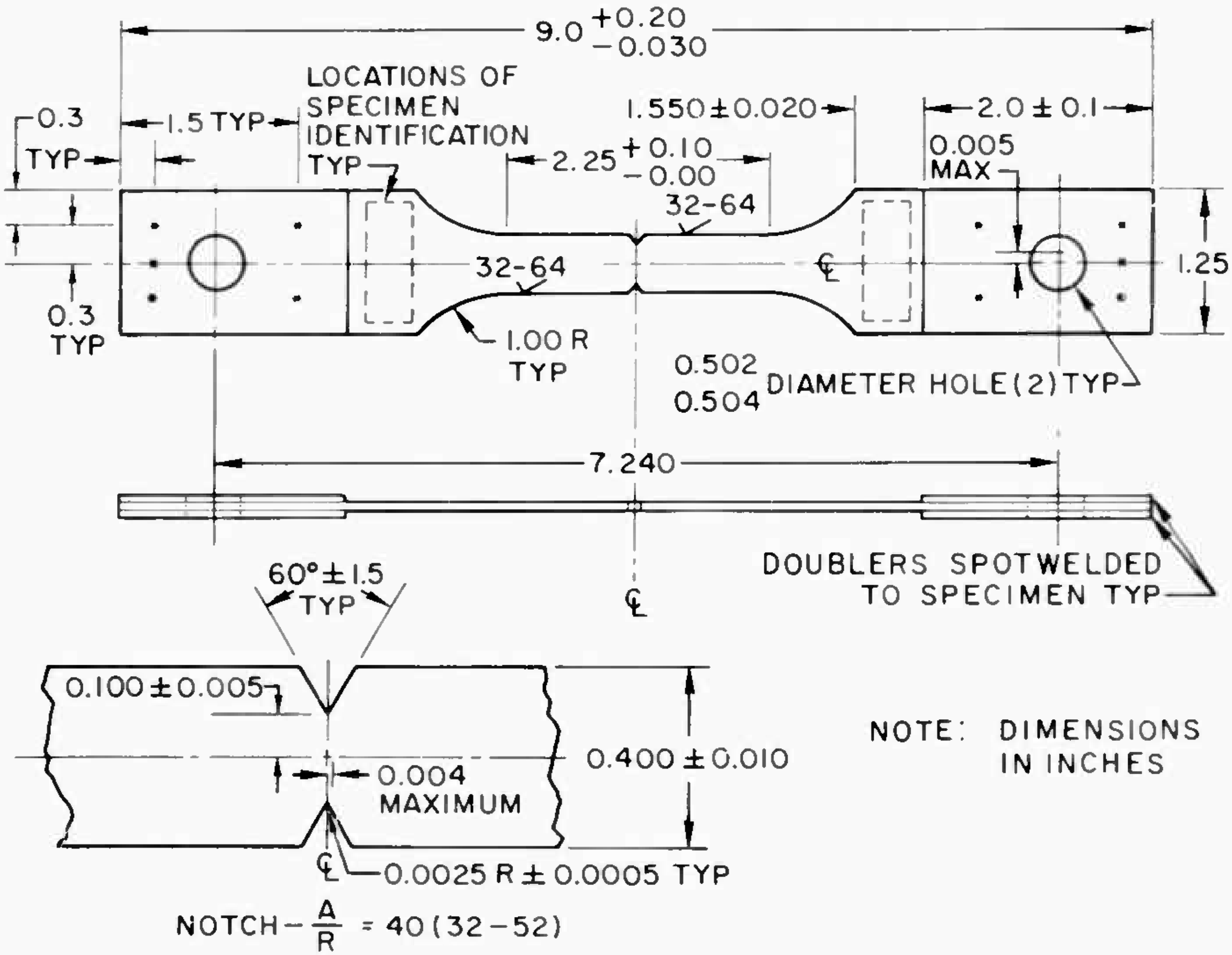

Fig. 4. Stainless Steel Test Specimen with K_t of 6.3

Table 3. Correlation of Notched-to-Unnotched Tensile Ratios with Fatigue Properties*

	Test Temperature	Notched-to-Unnotched Tensile Ratio (K_t = 6.3)		Cycles to Failure† 0 to 140,000 Psi
		Longitudinal	Transverse	
Type 301, 60% cold rolled	+78° F	1.05	0.97	934 (*a*)
	−320	0.99	0.87	2671 (*a*)
	−423	0.92	0.68	633 (*a*)
Type 310, 75% cold rolled	+78	1.07	0.97	—
	−320	1.11	1.07	1855 (*c*)
	−423	1.12	1.03	2000‡
Type 301-N, 60% cold rolled	+78	1.08	0.97	632 (*b*)
	−320	0.84	0.80	327 (*b*)

*Tests were made on weld joints 3 1/2 in. wide, 20 in. gage length, 38 in. over-all length. All sections 0.020 in. thick.

†For fatigue tests, weld joints were axially loaded and unloaded between 0 and 140,000 psi in tension at 6 cycles per min. Letter code indicates average of (*a*) three tests; (*b*) five tests; (*c*) two tests.

‡Test stopped after 2000 cycles. One small crack was evident in heat-affected zone of one of the resistance welds in outer row of spots. Estimated fatigue life: 3000 cycles.

The authors (Refs. 23-26) contend that when the notched-to-unnotched tensile ratio was substantially unity or higher, the fatigue resistance was high and increased with decreasing test temperature. This is because the tensile strength of the joints increased with decreasing temperature; thus, 140,000 psi became a lesser proportion of the tensile strength, with a corresponding increase in fatigue resistance. When, however, the notched-to-unnotched ratio fell below unity, indicating the initiation and propagation of fracture at less than tensile strength stress levels, the fatigue resistance declined considerably - in the case of type 301 steel, declining to 630 cycles at $-423^{\circ}F$ from a level of 2670 cycles at $-320^{\circ}F$ with a drop in notched-to-unnotched tensile ratio of 0.99 to 0.92, and, in the case of the nitrogen modified type 301 steel, declining from 680 cycles at $78^{\circ}F$ to 430 cycles at $-320^{\circ}F$ with a drop in notched-to-unnotched ratio from 1.08 to 0.84.

When notched tension tests were performed with specimens having a K_t value of 3, it was not possible to discriminate among these steels; all exhibited approximately the same high notched-to-unnotched tensile ratios. Also, the authors claimed that at the other extreme, other work (Ref. 28), which evaluated the notched tensile properties of severely cold-rolled type 301 stainless steel sheet with a sharp notched tension specimen having a K_t of approximately 17, showed these materials to possess low notched-to-unnotched ratios in the range of 0.5 to 0.6.

These experiences, coupled with other data on aluminum and titanium sheet alloys as well as on heat treatable stainless steels, resulted in visualizing the effect of notch severity upon the fracture characteristics of sheet alloys as shown in Fig. 5. At low stress concentration values (K_t of 2 to 3), both ductile and brittle materials may exhibit the same high notched-to-unnotched tensile ratios; thus, these tests fail to be discriminatory in predicting whether structures fabricated from the materials will perform satisfactorily or will be subject to catastrophic, brittle fracture. While tests with a K_t of 17 do permit judgments to be made as to the relative toughness or brittleness of materials, the effect of high stress concentrations is to make all materials

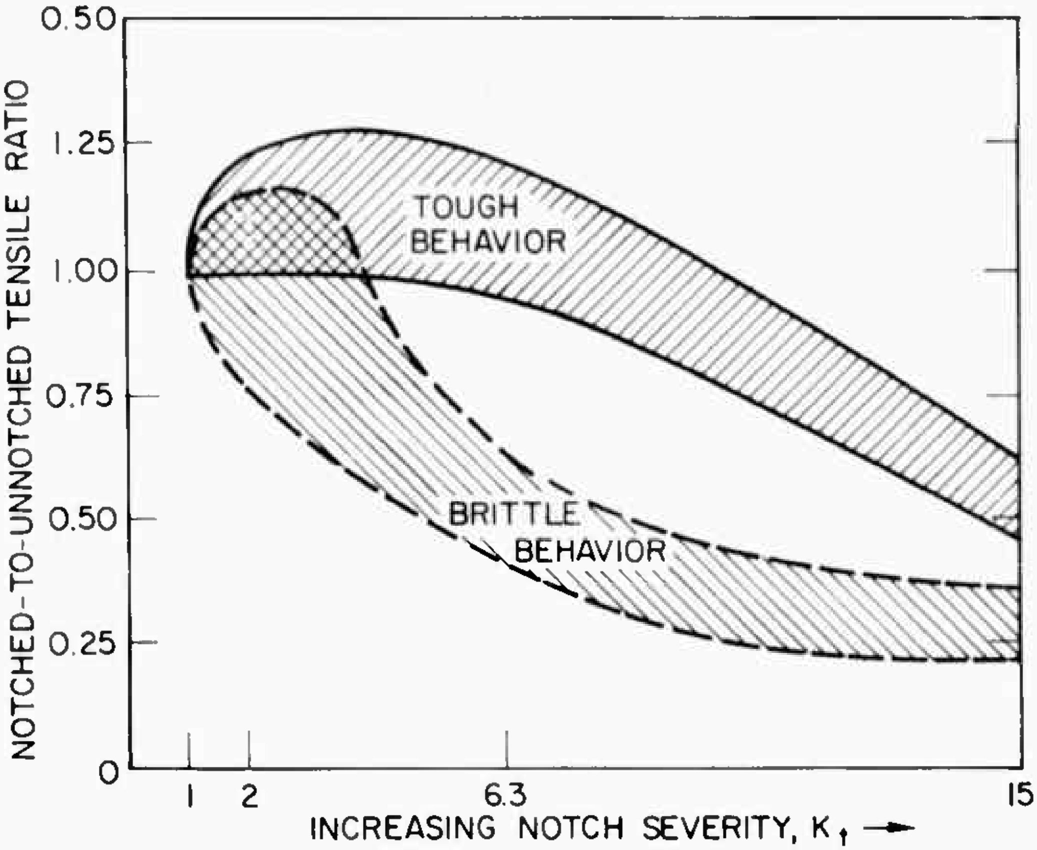

Fig. 5. Effect of Stress Concentration Factor upon Notched-to-Unnotched Ratios of Tough and Brittle Materials

behave in a relatively brittle manner; a brittle material may exhibit a ratio of 0.3 to 0.4 while a tough material, a ratio of 0.6 to 0.7. Thus, some of the discrimination is lost by the extreme severity of the test. At some intermediate notch severity range (K_t of 6 to 8) there seems to be maximum discrimination between tough and brittle materials so that, at least based upon the correlated notched-to-unnotched tension tests and simulated service tests conducted on actual structures, it is possible to assess the relative merits of candidate materials with greater reliance because of the greater spread in the ratios between tough and brittle materials.

In a rebuttal to this stand, Brown and Jones (Ref. 29) claim that attempts to correlate fatigue behavior with results of static notched tests should be treated with caution. They also contend that this has not in fact been properly accomplished citing that in the case of the 301-60% CR the decrease in the notch/tensile ratios is insignificant and is well within the limits of scatter. Among the points urged by Brown and Jones are (1) that fatigue life increase (for 301 steel in going from 78 to 320°F) concurrent with notch/tensile ratio decrease indicates lack of correlation, and (2) that three tests of the 310 stainless steel (one unfailed) are insufficient evidence to support correlation. In addition, Brown and Jones present data showing that the notch/tensile ratio of 310 decreased with decreasing temperature down to -423°F in contrast to the data of Watson, et al., (Ref. 23).

In answer to this criticism, Watson, et al., (Ref. 30) contend that their approach is sufficient to provide engineering data and was not intended to be a contribution to the theoretical development of the field of fracture toughness. In defense of the fatigue-notch tensile correlation, Watson, et al., presented additional data on 310, which showed no decrease in fatigue life along with no change in notch ratio from 78 to -423°F, somewhat substantiating their hypothesis.

In a more recent work, Kaufman (Ref. 31) proposes the use of a notch/yield ratio (ratio of notch tensile strength to tensile yield strength) to be a more useful criterion for rating the relative notch sensitivities of alloys than the notch/tensile ratio. In his research performed on aluminum alloys, he shows that, in contrast to the case of notch/tensile ratios, the use of the notch/yield ratio (at $K_t > 10$) leads to essentially the same ratings of the alloys regardless of notch design. The ratings of the various aluminum alloys tested on this basis are shown in a table in Section VII.

Two important comments have been made concerning Kaufman's work. Sessler (Ref. 32) points out that for many materials the yield strength measurement is more sensitive to strain rate than is the tensile strength measurement and, thus, a constant strain rate should be used for determining yield strength if a meaningful comparison of alloys is to be accomplished. This should also be the case for determining the notch strengths. In addition, Watson (Ref. 33) confirmed the usefulness of the notch/yield ratio in the evaluation of Inconel-X. For a K_t of 6.3, notch/tensile values fell from 0.93 at +78°F to 0.86 at -423°F. For the same K_t, on the other hand, notch/yield values dropped from 2.15 to 2.00 which was considered a better reflection of the excellent low-temperature toughness of the material than the notch/tensile values. This better reflection is in part attributed to the fact that the notch/yield ratio is more significant for low yield/tensile strength ratio materials.

As Watson concludes, and is seconded by most investigators, a more thorough consideration of the fracture toughness of an alloy at low temperatures must include the absolute value and trend of the notched tensile strength, the values and trend of the notch/tensile ratio and notch/yield ratio, microstructure, fracture appearance, and correlation with scaled model tests if possible.

Finally, when comparing notched tensile data of any type, reported by different investigators, it is imperative that a comparison be made of the stress-concentration factors.

IV. COMPATIBILITY

An important consideration in selecting a container material within which to store the cryogenic liquid is the compatibility of this material with the stored liquid. Materials that are susceptible to catastrophic chemical reaction with the powerful oxidizers cannot be used for thin-walled, liquid-fueled vehicles. Therefore, because of this danger of violent oxidation reaction, the number of materials suitable for containing liquid oxygen or liquid fluorine is limited. Christian, et al.(Ref. 34) describe some of the results of tests in this area.

Titanium and aluminum are desirable structural materials because of their high strength-to-density ratios; however, they are also extremely chemically reactive. By suitable initiation, each of these metals will burn in oxygen with a highly exothermic reaction.

Tests were conducted to demonstrate whether a catastrophic titanium-oxygen reaction would result from several possible accidental occurrences when gaseous or liquid oxygen was contained in a thin-walled titanium tank. In each instance, the titanium sheet was fractured to expose a clean unoxidized surface while in contact with the liquid or gaseous oxygen. The evaluations included (a) pressurized diaphragm puncture tests, (b) tensile fracture tests of welded joints, and (c) simulated micrometeroid puncture tests on two titanium alloys, Ti-5Al-2.5Sn and Ti-6Al-4V. In addition, comparison tests were made on type 301 stainless steel (extra full hard) and 2024-T3 aluminum.

A significant fact disclosed by these tests is that the titanium-oxygen reaction appears to be initiated only by rapid fracture. Slowly propagating fractures caused by cyclic loads do not cause the reaction, but a rapid tensile fracture or puncture of a thin sheet diaphragm in contact with oxygen generally does. It is likely that minute metal particles, separating from the rapidly fracturing surfaces, are immediately oxidized (similar to the "sparking" of abraded metal) with the release of sufficient heat energy to initiate burning. In addition, adiabatic heating of rapidly fractured surfaces can initiate the oxidation reaction.

Neither stainless steel nor 2024 aluminum react when subjected to rapid fracture. Aluminum has been reacted with liquid oxygen in the standard falling-weight compressive impact test; however, its susceptibility to reaction is much less than for titanium.

A number of coatings for titanium, generally oxidation inhibitors, have also been evaluated. While a small decrease in the degree of the reaction was noted, the coatings did not constitute a sufficient preventative. Thus, the relatively high susceptibility of titanium alloys to catastrophic reaction eliminates them from consideration for structural service as thin sheet in contact with liquid or gaseous oxygen.

These results are in agreement with the conclusions obtained in a somewhat more extensive compatibility study reported by Riehl, Key, and Gayle (Ref. 35). In these tests, titanium displayed high reaction sensitivity to compressive impact in the presence of liquid oxygen, but both electroless nickel and electroless copper applied to the surface of the sheet prevented the reaction. These coatings did not prevent the reaction when pressurized vessels containing liquid oxygen and made of thin titanium sheet were punctured by bullets, darts, or pins.

Titanium alloys are also sensitive to the oxygen reaction when initiation is from a detonating cap or from a high energy spark. Aluminum can be reacted only by a very heavy explosive shock. Neither titanium, aluminum, nor stainless steel was sensitive to an oxygen reaction in tests involving initiation by vibration, pressure cycling, sonic energy, or rupture of a thin wall container by excessive pressure.

Liquid hydrogen is practically inert to all metals and alloys. It exhibits extreme wettability, and recent research (Ref. 36) has determined that contact angles (θ) for the alloys studied were all zero degree. In general, liquid hydrogen's behavior in contact with glass or steel is similar to that of an organic liquid such as benzene.

The development of the Pratt and Whitney RL-10 liquid rocket engine is described in Ref. 37. No problems with the liquid hydrogen from the standpoint of corrosion and handling are noted.

A recent materials review for a LOX-LH_2 aerospaceplane (Ref. 38) contains results of a study of a titanium alloy for the LH_2 tankage. In thermal exposure tests in a gaseous hydrogen atmosphere at temperatures up to $800^{\circ}F$, it was discovered that the subsequent notch/tensile strength at $-423^{\circ}F$ was lowered. This was attributed to diffusion of hydrogen into the titanium, which was confirmed by the presence of hydride platelets in the microstructure.

Liquid fluorine, on the other hand, is an extremely aggressive liquid and attacks practically all metals and alloys to various degrees. The presence of either water or organic material in contact with the liquid fluorine is particularly critical because products such as HF and OF_2 result therefrom. These products, in turn, readily attack the container materials.

Alloys which have shown some degree of preference over others include monel, 304 and 347 stainless, "A" and "L" nickel, inconel, some Mg alloys, tantalum, Zr-2, and SAE 1010 steel. Cleanliness of fluorine systems cannot be overemphasized. It is also recommended that prior to contact with liquid fluorine the metal surfaces be passivated by a treatment with gaseous fluorine.

Liquid nitrogen and helium, like hydrogen, can be considered inert and present no corrosion problems.

V. ENGINEERING ALLOYS

In spite of some disagreement among investigators concerning the nature of notched tension testing and its interpretation, most will agree on the relative rating of alloys for a specific application. Table 4 has been reproduced from Metal Progress (Ref. 39) and presents data ranging from 78 to -423°F for all alloys showing any promise as structural high strength/weight ratio materials at low temperatures. The data are based upon the heretofore discussed K_t value of 6.3. Although questioned as to its merit, the set of data is probably the most extensively established under a constant specimen geometry and K_t factor, available at this time. Equally important, from an engineering standpoint, the data have been used as a point of reference in the Atlas and Centaur programs. In addition, the data have been partially responsible for the successful development of the excellent low-temperature alloy, Ti-5Al-2.5Sn (ELI).

It has been shown that metals having the FCC structure possess the best low-temperature properties. Further, those having the close-packed structure such as CPH titanium, zirconium, and hafnium would also have good low-temperature properties. This has been substantiated by the family of alloys now being utilized as low-temperature structural materials. The austenitic stainless steels, aluminum alloys, and nickel alloys all have the FCC structure, while the titanium alloys have the CPH structure. The individual merits of these alloys for cryogenic service are discussed in later sections.

Table 4. Tensile and Notched Tensile Properties of Alloys at Various Temperatures

Alloy	Test Temperature, °F	Yield Strength*, Psi	Tensile Strength, Psi	Elongation, %	Ratio, Notched (K_t = 6.3) to Unnotched Tensile Strength
Type 202 stainless 55% CR†	78	174,000	212,000	6.0	1.07
	-320	273,000	316,000	4.0	0.72
	-423				<0.49
Type 301 stainless 60% CR	78	200,000	224,000	11	1.07
	-100	237,000	253,000	15	0.98
	-320	254,000	323,000	20	0.92
	-423	308,000	335,000	3.5	0.90
Type 301N stainless 60% CR	78	200,000	223,000	12	1.08
	-320	244,000	340,000	18	0.84
	-423	297,000	334,000	12	0.78
Type 302 stainless 60% CR	78	178,000	206,000	3.0	1.08
	-320	228,000	307,000	29	0.92
	-423	249,000	294,000	20	0.95
Type 304L stainless 50% CR	78	158,000	176,000	6.0	1.09
	-100	186,000	198,000	5.0	1.09
	-320	187,000	251,000	33	1.04
	-423	231,000	278,000	1.0	1.09
Type 310 stainless 25% CR	78	157,000	181,000	2.0	1.07
	-100	190,000	204,000	3.0	1.08
	-320	223,000	251,000	10	1.11
	-423	261,000	290,000	5.0	1.12
A-286 stainless aged at 1325 F	78	98,900	150,000	15	0.98
	-320	122,000	203,000	23	0.88
	-423	139,000	233,000	18	0.87
AM-355 stainless CRT‡	78	278,000	297,000	5.4	0.85
	-100	287,000	308,000	17	0.89
	-320	328,000	353,000	9.5	0.46
	-423	329,000	347,000	0	0.34
"K" Monel aged at 1080 F	78	97,300	154,000	22	0.83
	-100	107,000	166,000	24	0.93
	-320	120,000	183,000	30	0.95
	-423	136,000	200,000	28	0.99
Inconel X-750 aged at 1300 F	78	118,000	174,000	26	0.97
	-100	122,000	189,000	30	0.92
	-320	130,000	214,000	31	0.86
	-423	134,000	233,000	30	0.85
René 41 double aged at 1950 and 1400 F	78	138,000	181,000	18	0.91
	-100	148,000	192,000	13	0.90
	-320	161,000	202,000	9.0	0.94
	-423	178,000	212,000	6.0	0.99
Haynes 25 20% CR	78	126,000	164,000	16	1.03
	-100	146,000	192,000	16	1.00
	-320	181,000	255,000	23	0.91
	-423	208,000	268,000	20	0.98
Hastelloy B 40% CR	78	177,000	191,000	3.0	1.15
	-100	207,000	222,000	5.0	1.10

Alloy	Test Temperature, °F	Yield Strength*, Psi	Tensile Strength, Psi	Elongation, %	Ratio, Notched (K_t = 6.3) to Unnotched Tensile Strength
Hastelloy B 40% CR	-320	208,000	228,000	12	1.16
	-423	240,000	283,000	16	1.09
Aluminum					
2014-T6	78	65,700	73,100	11	1.02
	-100	69,300	76,400	12	1.04
	-320	74,400	87,100	14	0.98
	-423	86,200	104,000	17	0.94
2012-T4	78	37,500	61,000	17	0.92
	-100	38,500	62,100	19	0.94
	-320	48,000	82,400	21	0.87
	-423	65,000	99,800	19	0.85
2024-T4	78	42,800	67,700	19	0.87
	-100	43,700	69,000	22	0.87
	-320	54,100	84,000	27	0.85
	-423	73,300	107,000	16	0.83
2219-T87	78	58,200	70,700	9	0.99
	-100	62,400	76,400	9	0.98
	-320	69,200	88,400	11	0.97
	-423	76,400	104,000	14	0.92
5052-H38	78	40,000	45,100	7.0	1.07
	-100	41,000	47,000	11	1.07
	-320	48,000	82,600	25	1.01
	-423	54,700	89,700	32	0.90
5083-H38	78	56,700	62,700	5.1	0.99
	-320	65,000	82,100	15	0.94
	-423	71,500	101,000	13	0.86
5086-H34	78	35,700	47,800	9.4	1.02
	-100	36,600	48,000	15	1.03
	-320	40,000	65,400	24	0.95
	-423	47,000	95,300	30	0.75
5154-H38	78	40,200	47,400	9.2	1.04
	-100	40,800	49,300	14	1.04
	-320	47,100	66,200	30	0.97
	-423	54,000	93,500	35	0.93
5456-H343	78	47,200	58,600	7.1	0.92
	-100	47,800	59,000	8.2	0.84
	-320	53,000	72,700	8.4	0.68
	-423	60,400	83,500	5.9	0.66
6061-T6	78	43,500	47,400	11	1.05
	-100	45,000	51,700	11	1.04
	-320	51,200	62,300	16	1.00
	-423	54,000	80,100	23	0.93
7025-T6	78	70,400	79,500	9.2	1.02
	-100	76,200	85,100	10	0.99
	-320	86,500	97,200	10	0.78

Alloy	Test Temperature, °F	Yield Strength*, Psi	Tensile Strength, Psi	Elongation, %	Ratio, Notched (K_t = 6.3) to Unnotched Tensile Strength
7075-T6	-423	100,000	116,000	2.2	0.73
2079-T6	78	68,000	72,100	11	1.08
	-100	73,200	81,500	12	1.04
	-320	82,100	94,300	16	0.97
	-423	91,100	112,000	7.8	0.70
2178-T6	78	82,700	89,000	12	1.02
	-100	87,800	95,200	11	0.81
	-320	98,800	106,000	4.2	0.54
	-423	111,000	123,000	1.8	0.51
Titanium					
AMS-4901B, commercially pure annealed	78	67,600	84,300	26	1.35
	-100	89,200	109,000	25	1.19
	-320	136,000	152,000	18	1.05
	-423	173,000	186,000	7.9	0.68
Ti-5Al-2.5Sn annealed	78	113,000	118,000	19	1.34
	-100	135,000	142,000	18	1.23
	-320	184,000	196,000	15	1.15
	-423	230,000	247,000	15	0.97
Ti-5Al-5Zr-5Sn annealed	78	121,000	126,000	17	1.23
	-100	145,000	150,000	12	1.21
	-320	192,000	209,300	12	0.95
	-423	235,000	262,000	11	0.70
Ti-6Al-4Zr-1V annealed	78	137,000	142,000	16	1.22
	-100	160,000	167,000	15	1.17
	-320	212,000	227,000	10	0.81
	-423	264,000	278,000	4.6	0.54
Ti-7Al-12Zr annealed	78	133,000	143,000	11	1.24
	-100	147,000	158,000	11	0.98
	-320	197,000	214,000	12	0.78
	-423	240,000	251,000	6.3	0.58
Ti-8Al-2Cb-1Ta annealed	78	131,000	142,000	12	1.19
	-100	156,000	165,000	13	1.20
	-320	204,000	217,000	13	1.12
	-423	250,000	265,000	1.3	0.86
Ti-8Al-1Mo-1V annealed	78	135,000	142,000	16	1.09
	-100	153,000	163,000	16	0.98
	-320	198,000	215,000	15	0.89
	-423	230,000	242,000	3.2	0.77
Ti-6Al-4V annealed	78	128,000	141,000	11	1.11
	-100	158,000	166,000	11	1.01
	-320	211,000	219,000	10	0.87
	-423	245,000	253,000	1.8	0.72
Ti-13V-11Cr-3Al annealed	78		135,000	22	1.35
	-320		288,000	0	0.56

*0.2% offset; †CR = cold rolled; ‡CRT = cold rolled and tempered

VI. STEELS

The austenitic stainless steels (type 300 Series), retaining the FCC structure of γ-iron, exhibit maximum notch toughness at extreme low temperatures. Considerable testing of these alloys for cryogenic applications has been reported (Refs. 23, 26, 28, 40, 41, and 42) and a summation of their properties is shown in Table 4. Although these alloys have low yield strengths in the fully annealed condition, severe cold work can increase the yield and tensile strengths to 160,000 and 200,000 psi minimum values respectively. Thus, in this condition they become candidate alloys for LOX and LH_2 tankage.

The effect of low temperatures on the mechanical properties of 301-60%CR, 301-78%CR, 304ELC-50%CR, and 310-75%CR is shown in Fig. 6. Type 301-60%CR(XFH) was selected for the LOX tanks on the Atlas vehicle. It exhibits a tensile strength that increases from about 220,000 psi at 78°F to 330,000 psi at -423°F and a yield strength that increased from 200,000 psi to 285,000 psi over the same temperature range. This steel when cold rolled 60 percent at room temperature becomes 60 percent martensitic in structure and then transforms to 100 percent martensite when tested in tension at -423°F. Being low in carbon content (0.1 maximum) and primarily Fe-Ni, this martensite exhibits greater toughness than the higher carbon content martensites in the medium alloy steels. Still, it is this transformation to 100 percent martensite, which no doubt accounts for the sharp decrease in elongation below -320°F. Consequently, this steel is not particularly recommended for liquid hydrogen service in the 60 percent cold rolled condition. Decreasing the degree of cold rolling would increase the steel's tolerance at -423°F but, dependent upon the amount, it still may be less than that at -320°F.

In an attempt to improve austenite stability at low temperatures, tests were performed on the same alloy with additions of 0.10 percent nitrogen (301-N). Although this alloy exhibited similar room temperature properties, lower

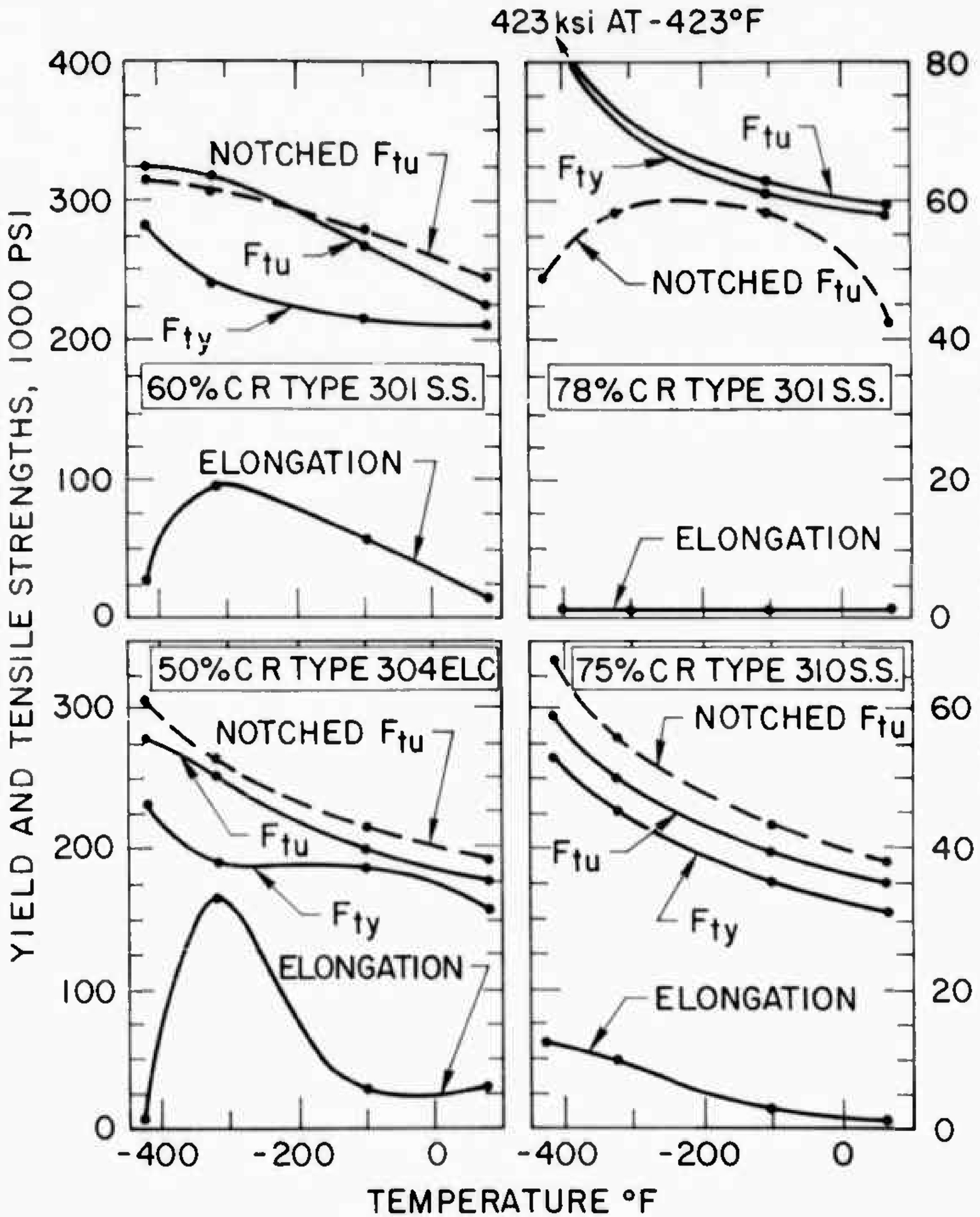

Fig. 6. Effect of Low Temperatures on Mechanical Properties of Various Stainless Steels

notch ratios were obtained at low temperatures due in part to structure instability resulting in transformation to martensite prior to straining.

On the other hand, type 310 (25Cr-20Ni), when 75 percent cold reduced at room temperature and strained to fracture at -423°F, completely retains the austenite. Although considerably lower in tensile yield strength at 78°F than the 301, it is almost equal to the 301 at -423°F and has a far superior notch ratio value. The stability in structure also accounts for a higher fatigue life at a given stress level than for any other 300 series stainless.

The effect of temperature on the notch/tensile ratios of four 300 series stainless steels are shown in Fig. 6. Since the maximum elongation (Fig. 7) and the minimum notch/tensile ratio occur at the same temperature (-320°F) for 304ELC, it is evident that percent elongation bears no relation to toughness. Also the notch/tensile ratio for the 301-78%CR is seen to vary widely with temperature, while the elongation remains constant over the same temperature range.

Campbell and Rice (Ref. 43) studied the low-temperature properties of the low alloy and heat-treatable stainless steels. AISI 4340, H11, 300M, AM 350, and the 17-7PH steels were all found to undergo a ductile/brittle transition at -100°F or above and therefore are not recommended for cryogenic service. On the other hand, the precipitation-hardened stainless steel A-286 has demonstrated toughness down to -423°F under moderate notch conditions.

The recent development, specifically for low-temperature use (Ref. 44), of a ductile iron casting alloy, is also worthy of mention. The composition of this alloy is 2.5% Si, 3.75% Mn (minimum), 21% Ni, 0.5%Cr (maximum), 2.6%C, and the balance is Fe. This alloy provides austenite stability down to -320°F and exhibits 20 ft-lb Charpy impact values at -320°F; it is recommended for pumps, valves, compressors, piping and fittings, and other cryogenic handling equipment.

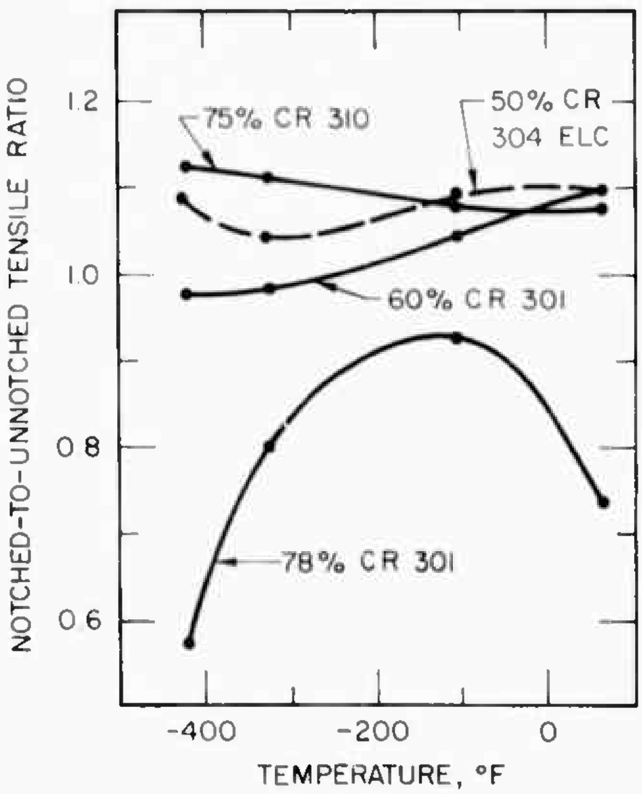

Fig. 7. Effect of Temperature on the Notch/Tensile Ratios of Four Stainless Steels

VII. ALUMINUM ALLOYS

Aluminum alloys have demonstrated the capability to provide excellent service at extremely low temperatures. Taking advantage of their basic face-centered-cubic (FCC) structure and solid solution strengthening, selected alloys in the 2000, 5000, and 6000 series have exhibited good toughness properties down to -423°F and are thus ideal for liquid oxygen and hydrogen tankage. Some of the work done in this area is reported in Refs. 45 through 51.

A summation of low-temperature properties of the candidate alloys can be found in Table 4. Properties of four selected alloys down to -423°F have been plotted by Hurlich (Ref. 40) and are shown in Fig. 8.

In a review paper (Ref. 49), Kaufman cites the excellent properties of the Al-Mg alloys (5000 series) for low-temperature service. It was alloys in this system that were first used for LOX tankage on the early missile vehicles and have carried through to the Saturn vehicle. Although readily weldable, ductile, and tough at low temperatures, they have low strengths which put them at a disadvantage for the later, more-optimized aerospace vehicles. As a result, the 5000 series alloys have given way to the higher strength 2000 series alloys.

The 2000 series alloys are age-hardenable and contain copper as their chief alloying constituent. Alloy 2014-T6 is being used for LOX tankage in the Thor and Titan I systems and is under consideration for LH_2 tankage in upper stages of the Saturn. Problems relating to weldability and properties of weldments have been limitations with this alloy. Within the past two years, a new alloy, 2219, has been gaining favor for cryogenic applications (Refs. 52 and 53). Considered more readily weldable than alloy 2014, alloy 2219 has slightly lower static mechanical properties.

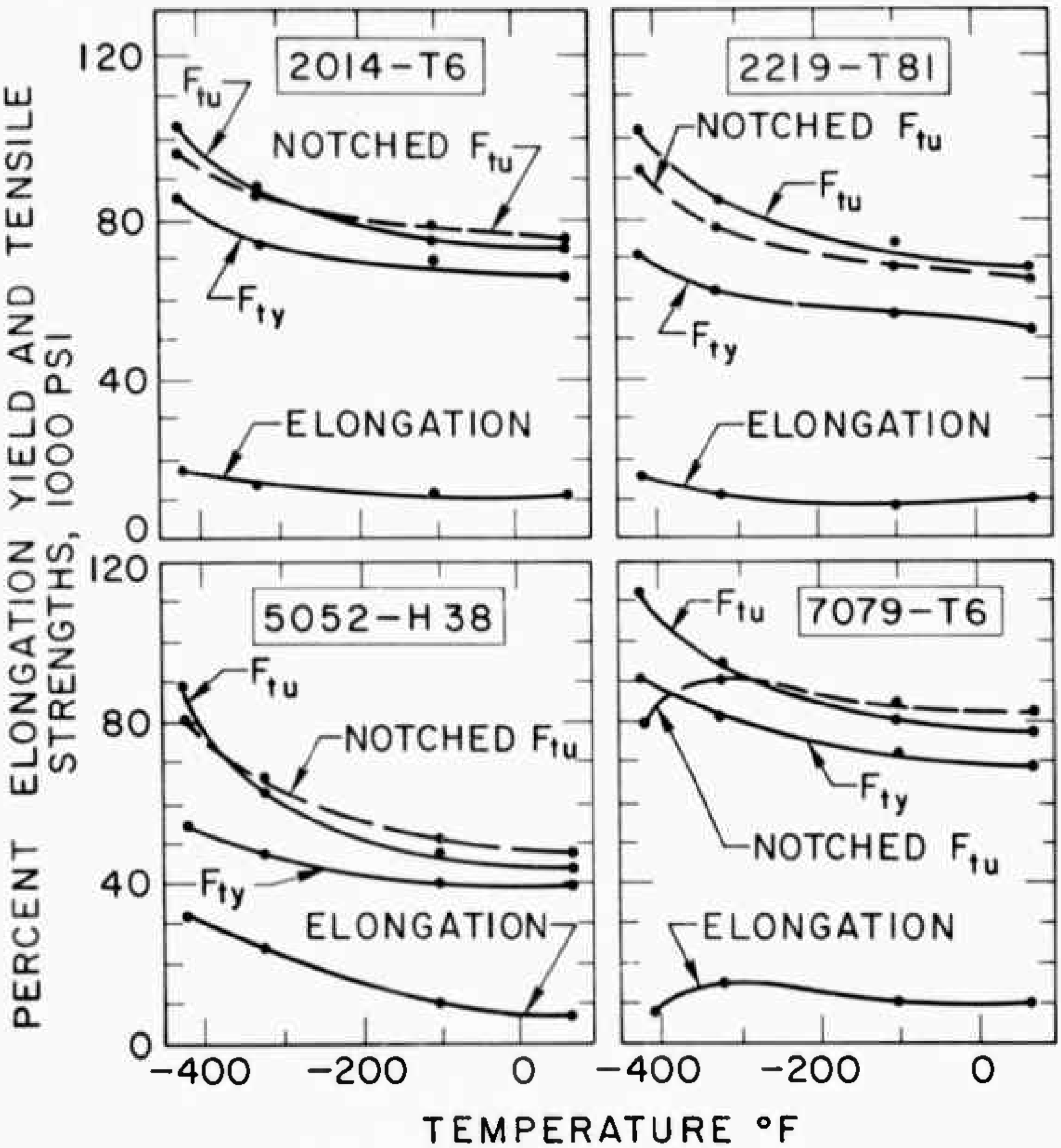

Fig. 8. Properties of Four Aluminum Alloys at Various Temperatures

The 7000 series alloys have the highest strengths of all aluminum alloys; however, their high degree of dispersion strengthening results in brittle behavior at low temperatures. They are not considered weldable and are not recommended for low-temperature use. Recent work, however, on experimental alloys in the 7000 series has shown that the new 7039 alloy has high notch toughness at -423°F (Ref. 54).

As mentioned previously, Kaufman (Ref. 31) made use of the notch/yield ratio for evaluating aluminum alloys. In a review paper, McClintock (Ref. 55), although replotting the notch/tensile and notch/yield data and obtaining a slightly different alloy rating, substantiates the claim that the notch/yield criterion gave better correlation.

The rating of aluminum alloys on a toughness basis down to -423°F by Hurlich, Kaufman, and McClintock is shown in Table 5. Although slight discrepancies exist (due in part to differences in K_t values and notch ratios) all investigators agree, in general, on the superiority of the 2000 and 5000 series alloys. This superiority is illustrated in Fig. 9, which shows the 7079 alloy notch ratio to decrease rapidly below -300°F.

Table 5. Toughness Ratings of Selected Alluminum Alloys at -423°F

**Hurlich (Ref.) (K_t 6.3)	*Kaufman (Ref.) (K_t-11)	*McClintock (Ref.) (K_t 11)	**McClintock (K_t-11)
2014-T6	5454-0	5454-0	6061-56
2024-T4	5456-0	5456-0	5154-H38
2219-T8	5454-H34	2219-T31	2219-T37
5052-H38	2219-T31	5454-H34	5454-H34
5083-H38	5154-H38	5154-H38	2219-T87
6061-T6	6061-T6	6061-T6	5454-H34
5086-H34	2219-T37	2024-T3	2219-T87
5086-H38	2024-T3	2219-T37	2219-T31
5154-H38	2219-T62	2219-T62	2219-T81
6061-T4	2219-T81	2219-T81	2024-T3
7079-T6	2219-T87	2219-T87	2014-T6
7075-T6	5456-H24	5456-H24	5454-0
7275-T6	2014-T6	2014-T6	5219-T62
7178-T6	2024-T86	7079-T6	2024-T86
	7079-T6	7075-T6	5456-H24
	7075-T6	X2020-T6	5456-0
	X2020-T6	7178-T6	5456-0
	7178-T6		7079-T6
			7075-T6
			X2020-T6
			7178-T6

* Nolch/yield basis
** Notch/tensile basis

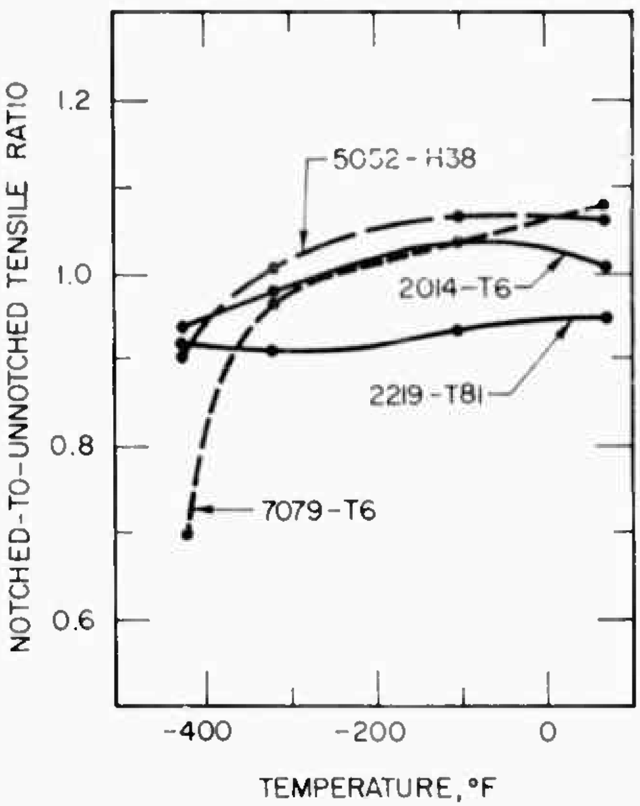

Fig. 9. Aluminum Alloy Tensile Ratios at Various Temperatures

VIII. TITANIUM ALLOYS

Titanium alloys' excellent low-temperature strength properties coupled with their low density make them extremely attractive for aerospace vehicle applications.

High strength-to-weight ratios, good formability and weldability, and excellent corrosion resistance have contributed to their selection as compressed gas storage vessels immersed in liquid nitrogen at $-320^{\circ}F$.

Results on extensive testing of titanium alloys at temperatures down to $-423^{\circ}F$ have been reported in the recent literature (Refs. 56-60).

Notched tensile test results for nine alloys are summarized in Table 4. High notch ratios are retained down to $-320^{\circ}F$ for eight of the alloys and to $-423^{\circ}F$ for the all alpha alloy Ti-5Al-2.5Sn. The well-established commercial alloy, Ti-6Al-4V, when heat-treated to the range of 160,000 psi, showed high toughness at $-320^{\circ}F$ and was selected for fabrication of the compressed helium gas bottles on the Atlas missile (Ref. 61). However, this alloy consists of the duplex $\alpha + \beta$ structure and below $-320^{\circ}F$ begins to become notch sensitive. A comparison of the notch/tensile values of Ti-5Al-2.5Sn, Ti-6Al-4V, 8Al-1Mo-1V, and 5Al-5Zr-5Sn down to $-423^{\circ}F$ is shown in Fig. 10. The notch ratios of these four alloys are plotted versus temperature in Fig. 11; the superiority of the 5Al-2.5Sn alloy, over the entire cryogenic range, is easily seen.

Although the full potential of titanium alloys for low-temperature use is now apparent, it has only been during the past three years that the picture has clarified. Many investigators (Refs. 56-60) found conflicting or inconclusive effects with the various alloys. It is now clear that the total residual impurity content, and in particular oxygen and iron, seriously affects the low-temperature toughness values.

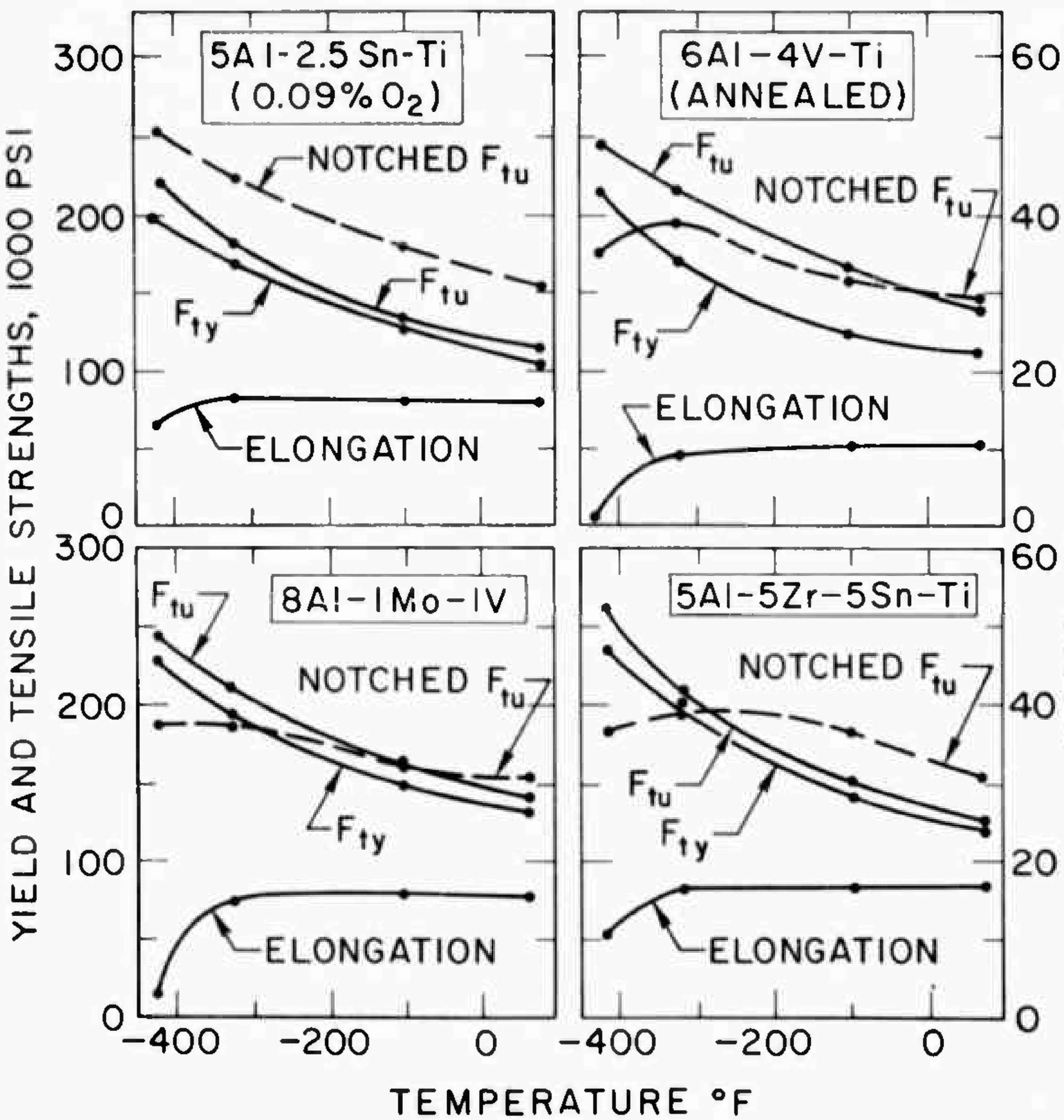

Fig. 10. A Comparison of Notch/Tensile Values for Four Titanium Alloys at Various Temperatures

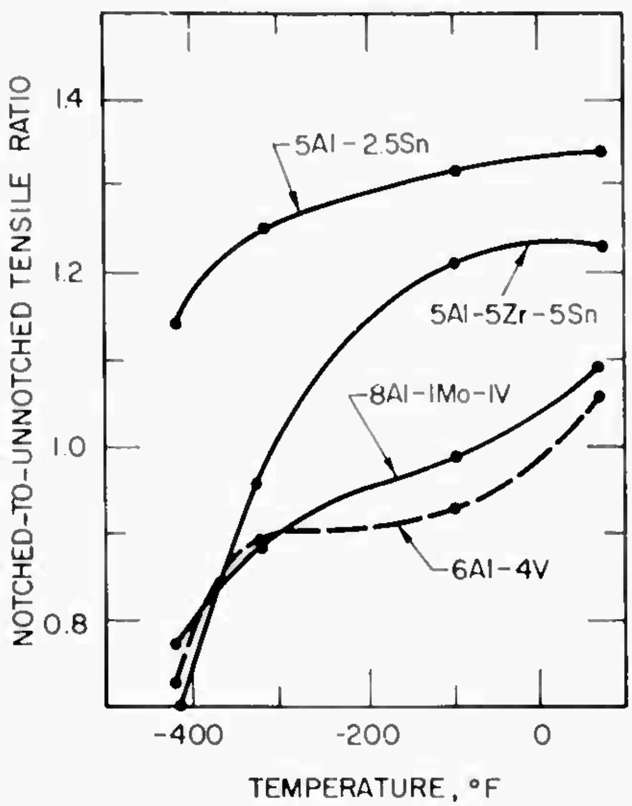

Fig. 11. Notch Ratios of Four Titanium Alloys vs Temperature

Schwartzberg (Ref. 59) determined that a total interstitial content of 0.107 percent for O_2, N_2, and C in 5Al-2.5Sn resulted in an 18 percent elongation at 20°K. Increasing this impurity total to 0.188 percent lowered the elongation to 1.2 percent.

Other recent works (Ref. 60, 62, 63) have attempted to delineate the individual effects of oxygen and iron in both the 5Al-2.5Sn and 6Al-4V alloys. Table 6 is a summation of data showing the variation in notch/tensile ratio with oxygen content for the two alloys with other impurities held constant. Table 7 is similar to Table 6 except that only the 5Al-2.5Sn alloy is considered and the variable is iron content. Results of simultaneous variation in both oxygen and iron contents are listed in Table 8. Figure 12 indicates graphically the variation of the tensile and notch ratio data with iron content (oxygen constant at 0.1 percent) for 5Al-2.5Sn alloy at three test temperatures and shows that, in order not to increase the notch sensitivity, the maximum iron content lies between 0.2-0.3 percent at both -320 and -423°F.

On the basis of these data, a specification has been established setting maximum limits for these impurities:

Alloy	Maximum Impurity, %		
	Oxygen	Hydrogen	Iron
5Al-2.5Sn ELI	0.120	0.125	0.250
6Al-4V ELI	0.130	0.125	0.250

Thus, this is a perfect demonstration of alloys being developed specifically for low-temperature applications. Other titanium alloys, essentially all alpha, have recently been developed and are listed in Table 8 with their low-temperature properties. Although these alloys show poor notch behavior, it is likely that these properties are due in part to high oxygen content as well as other impurities.

Table 6. Effect of Oxygen on Mechanical Properties of Two Titanium Alloys

Oxygen Content, %	Test Temperature, °F	Yield Strength*, Psi	Tensile Strength, Psi	Elongation, %	Ratio, Notched (K_t = 6.3) to Unnotched Tensile Strength
		Ti-5Al-2.5Sn			
0.11	78	102,000	112,000	16.5	1.38
	−320	168,000	181,000	16.0	1.26
	−423	206,000	229,000	15.0	1.11
0.15	78	105,000	116,000	17.5	1.35
	−320	174,000	186,000	15.0	1.24
	−423	214,000	230,000	11.0	1.01
0.17	78	107,000	118,000	16.5	1.40
	−320	177,000	191,000	15.5	1.22
	−423	225,000	237,000	12.5	0.90
0.24	78	117,000	123,000	16.5	1.39
	−320	188,000	201,000	15.0	1.17
	−423	239,000	251,000	9.0	0.78
		Ti-6Al-4V			
0.09	78	127,000	135,000	12.0	1.22
	−320	202,000	218,000	13.0	0.96
0.15	78	127,000	136,000	11.4	1.25
	−320	204,000	220,000	14.0	1.00
0.17	78	127,000	138,000	12.5	1.23
	−320	215,000	232,000	10.5	0.89

Data shown are averages of three or more tests. All tests made in rolling direction of sheet; thickness is 0.030 in.

*0.2% offset.

Table 7. Notched (K_t = 6.3) and Unnotched Tensile Properties of Ti-5Al-2.5SnELI, 0.09-in. Sheet at Various Temperatures for Various Iron Levels

Fe, %	TEST TEMPERATURE, °F	0.2% YS, KSI	UTS, KSI	ELONGATION, %	NTS, KSI	NTS/UTS	NTS/YS
0.14	+78	104	115	15	156	1.36	1.50
	-320	176	185	15	238	1.28	1.36
	-423	207	227	6	254	1.12	1.23
0.21	+78	103	112	15	153	1.37	1.49
	-320	175	183	5	236	1.29	1.35
	-423	211	229	5	254	1.11	1.20
0.29	+78	106	117	13	157	1.35	1.48
	-320	183	190	6	234	1.23	1.28
	-423	207	238	2	227	0.95	1.10
0.39	+78	113	124	13	161	1.30	1.43
	-320	187	195	6	238	1.22	1.27
	-423	221	235	4	209	0.90	1.04

Table 8. Effect of Oxygen and Iron Contents (All other Elements Controlled within Narrow Range)

OXYGEN	IRON	TEST TEMP °F	F_{ty}, psi	F_{tu}, psi	ELONGATION, %	NOTCHED TENSILE STRENGTH, psi (K_t = 6.3)	NOTCHED-TO-UNNOTCHED TENSILE STRENGTH RATIO
0.07	0.15	−423	211,000	232,000	14.3	233,000	1.00
0.08	0.50	−423	226,000	252,000	6.3	180,000	0.71
0.20	0.27	−423	256,000	265,000	7.0	166,000	0.65

ALL TESTS IN LONGITUDINAL DIRECTION AND REPRESENT AVERAGE OF DUPLICATE SPECIMENS. GAGES OF SHEET WERE IN RANGE OF 0.035 TO 0.045 IN. AND SHEET WAS ROLLED FROM LABORATORY SIZE MELTS.

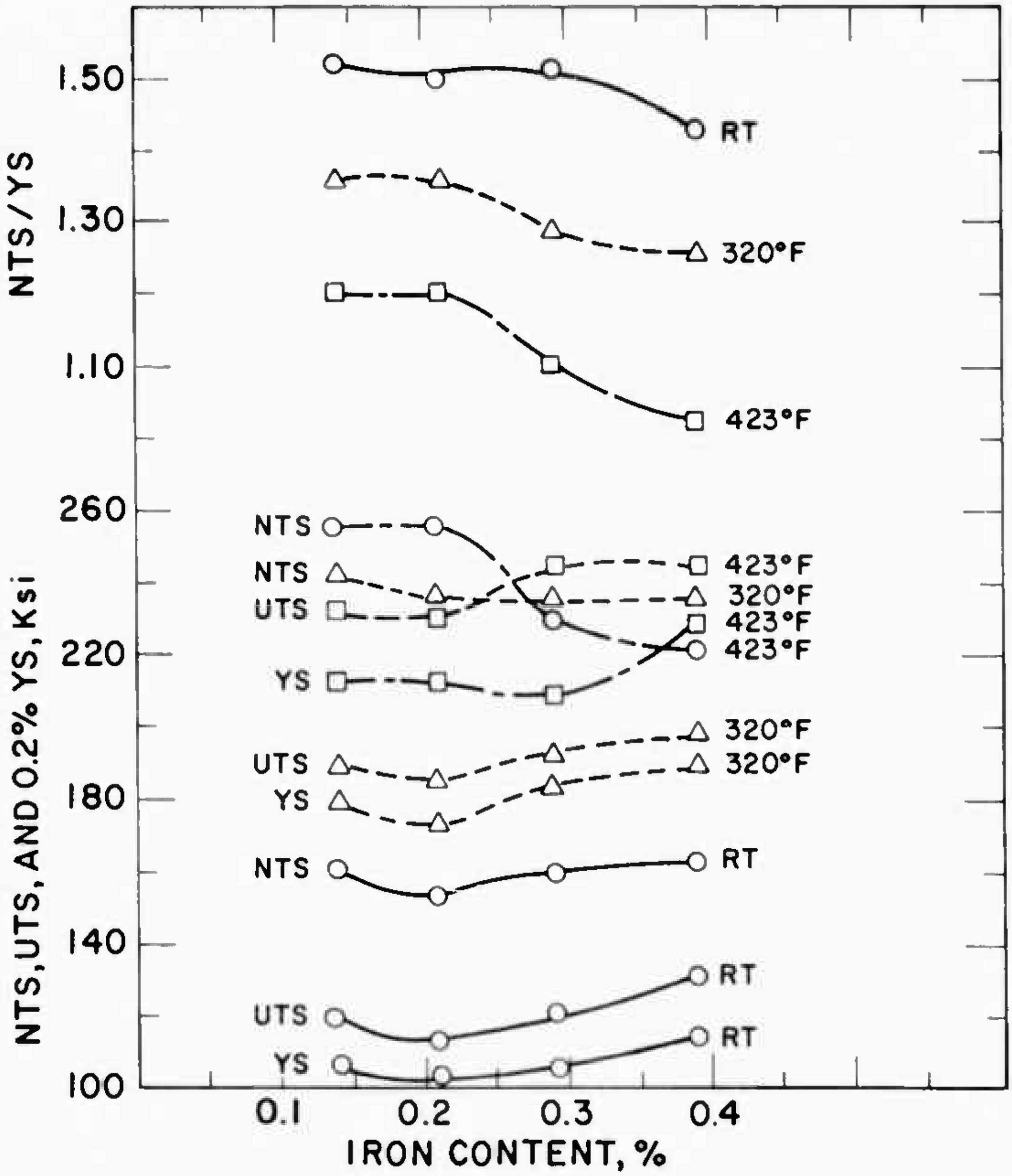

Fig. 12. Effect of Iron Content on Notched (K_t = 6.3) and Unnotched Tensile Properties of Ti-5Al-2.5SnELI at Various Temperatures

IX. NICKEL- AND COBALT-BASE ALLOYS

Nickel-base and cobalt-base alloys, possessing the FCC structure, have exhibited good low-temperature properties coupled with excellent high-temperature properties. This combination makes them attractive for cryogenic fueled vehicles having re-entry flight requirements. Their properties, down to -423°F, are described in a recent work by Watson and Christian (Ref. 64) and are shown in Table 4.

The effect of low temperatures on the mechanical properties of four nickel base alloys, K-Monel, Inconel-X, Rene'41, and Hastelloy B-40% CR, is shown in Fig. 13. Although this class of alloys shows the least amount of increase in yield and tensile strengths going from +78 to -423°F, the K-Monel and Inconel-X exhibit a rather high constant elongation over this temperature range. This excellent ductility has made these materials ideal for LOX ducting on the cryogenic fueled vehicles.

Figure 14 is a plot of notch/tensile ratio versus temperature for these alloys. Although Inconel-X shows a relatively low absolute value (0.85), it should be recalled from the previous discussion on notch toughness that the notch/yield ratio might possibly be a better criterion of rating these alloys. The notch/tensile ratio does not appear to be completely indicative of the behavior of alloys that have a low yield/tensile strength ratio and exhibit large amounts of plastic strain and necking prior to failure. The fibrous, 100 percent shear-type fracture and the increase in notch tensile strength tend to demonstrate the toughness of Inconel-X at -423°F.

Haynes 25, a cobalt-base alloy showed excellent ductility and toughness at -423°F in the 20 percent cold rolled condition but became brittle when the cold reduction was increased to 40 percent.

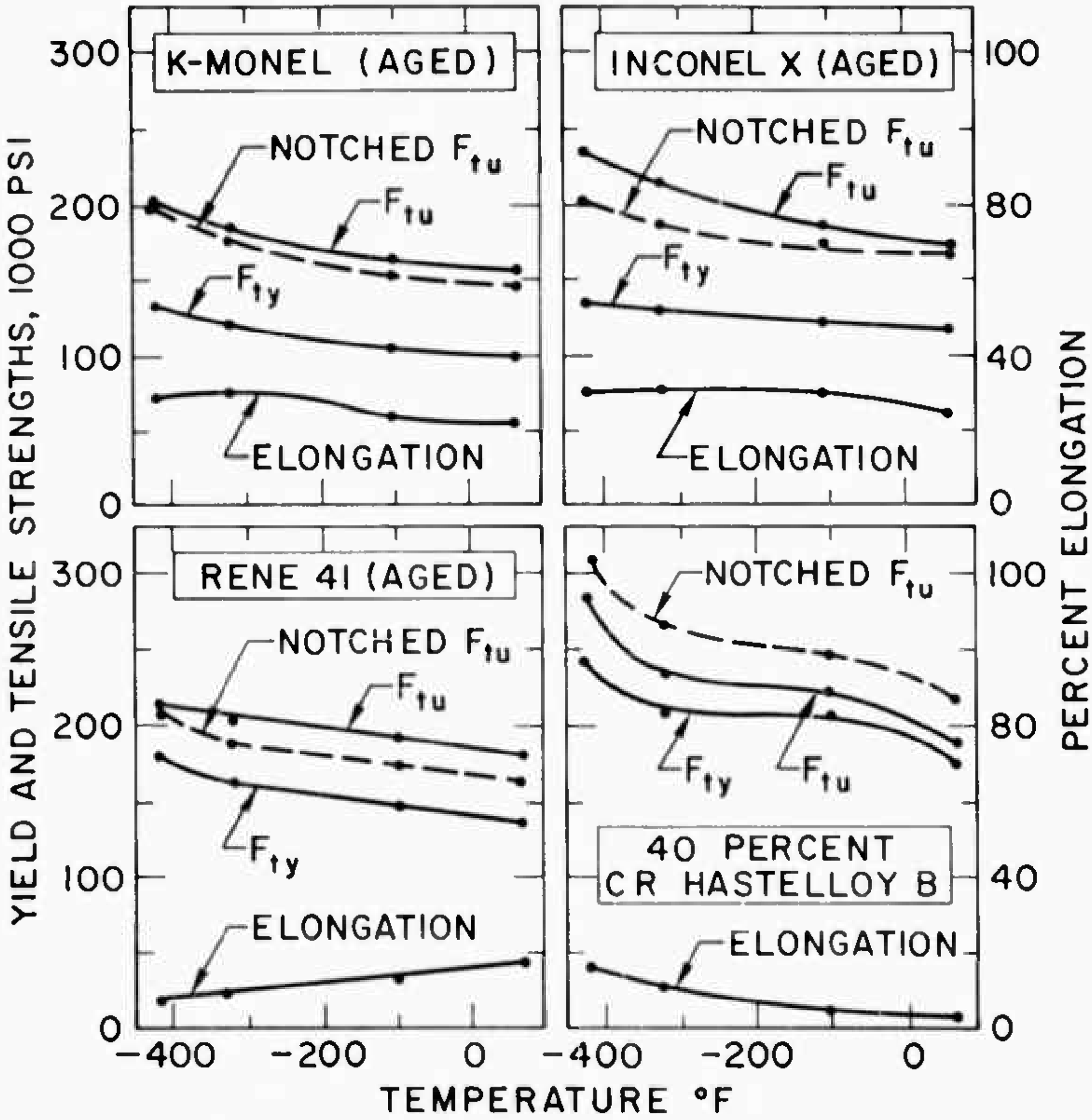

Fig. 13. Effect of Low Temperatures on the Mechanical Properties of Four Nickel-Base Alloys

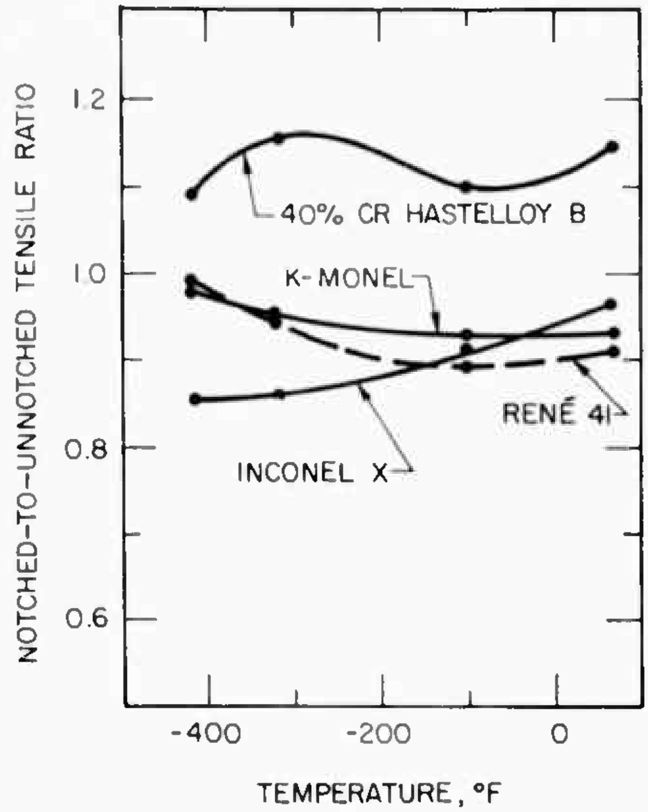

Fig. 14. Notch/Tensile Ratio vs Temperature for Four Nickel-Base Alloys

X. PROPERTIES OF WELDMENTS

The important criteria in selecting a material for use in missiles and space vehicles are its weldability and the mechanical properties of weldments (Refs. 26, 39, and 41). This is especially true of tanks containing liquid propellant since pressurization of the tank is required for fuel flow and in many instances for structural reasons. Also, all of the welded joints for propellant tanks and fuel lines must be gas-tight. The most common weld is a butt fusion weld. However, resistance roll seam and spot welds are also used in tank construction and are generally used to attach brackets.

Table 9 presents mechanical properties of fusion and resistance welds in several alloys tested at room and at very low temperatures. The only alloy with 100 percent joint efficiency (fusion weld) is Ti-5Al-2.5Sn. This titanium alloy was annealed, whereas the other alloys were cold rolled or in the age hardened condition. Joint efficiencies are less than 100 percent for the latter alloys because the weld metal and heat-affected zones are annealed and thus lower in strength.

In general, two methods are used in the aerospace industry for strengthening fusion welds to 100 percent joint efficiencies. One is the use of lands, obtained by chemical or machine milling. In the second, the reinforcement method, additional thicknesses of strip material are placed over the fusion weld and attached by resistance spot welds on either side. The propellant tanks of the Atlas and Centaur, which use type 301 and 310 stainless, extra hard cold rolled, to contain LO_2 and LH_2, respectively, are fabricated by the latter method. Values for cross tension and tensile shear strength of individual resistance spot welds are given in Table 9. The tension/shear ratios in the table determine the acceptability of a weld for structural use, a value of 0.25 or above being considered acceptable.

Table 9. Properties of Fusion and Resistance Welds at Various Temperatures

Alloy and Specimen	Test Temperature, F	Tensile Strength, Psi	Joint Efficiency, %
Type 301 stainless 60% CR† 0.025 in.	78	175,000	78
	−100	216,000	85
	−320	298,000	92
	−423	202,000	60
Type 304L stainless 50% CR 0.012 in.	78	78,000	44
	−100	144,000	72
	−320	216,000	89
	−423	250,000	91
Type 310 stainless 75% CR 0.020 in. 0.020 in.	78	86,700	48
	−100	109,000	54
	−320	162,000	64
	−423	208,000	71
AM-355 stainless CRT 0.032 in.	78	222,000	75
	−100	289,000	94
	−320	271,000	77
	−423	142,000	41
2014-T6 aluminum 0.063 in. (2319 filler wire)	78	53,100	73
	−100	56,700	74
	−320	61,900	71
	−423	75,600	73
Ti-5Al-2.5Sn annealed 0.032 in.	78	121,000	100
	−100	142,000	100
	−320	192,000	98
	−423	233,000	94

Resistance Spot Welds

Alloy and Specimen Thickness	Test Temperature, F	Cross Tension Breaking Load, Lb	Tensile Shear Breaking Load, Lb	Tension/Shear Ratio
Type 301 stainless 60% CR† 0.025 in.	78	662	1052	0.63
	−100	593	1281	0.46
	−320	160	1041	0.15
	−423	143	821	0.17
Type 304L stainless 50% CR 0.012 in.	78	256	409	0.63
	−100	242	510	0.47
	−320	265	634	0.42
	−423	306	666	0.46
Type 310 stainless 75% CR 0.020 in.	78	509	744	0.68
	−100	562	871	0.65
	−320	582	1096	0.53
	−423	533	1224	0.44
AM-355 stainless CRT 0.032 in.	78	851	1529	0.56
	−100	298	1758	0.17
	−320	186	903	0.21
	−423	162	858	0.19
2014-T6 aluminum 0.063 in. (2319 filler wire)	78	496	1179	0.42
	−100	509	1254	0.41
	−320	534	1332	0.40
	−423	480	1271	0.38
Ti-5Al-2.5Sn annealed 0.032 in.	78	360	1381	0.26
	−100	256	1670	0.19
	−320	268	1670	0.16
	−423	251	1587	0.16

*Inert gas, butt fusion welds with no filler metal, except as noted; †CR=cold rolled; ‡CRT=cold rolled and tempered.

Table 10 presents high-stress, low-cycle axial fatigue data on complex welded joints. These joints, except for the 2014 aluminum, were composed of butt fusion welds plus reinforcements, which were attached by four rows of resistance spot welds on either side of the fused joint. The specimen used was 38-in. long and had a test section 4-in. wide by 16-in. long.

The stress levels in the table are 75, 85, and 95 percent of the base metal yield strength or static strength of the joint, whichever is less, at each corresponding test temperature. Generally, a minimum of 200 cycles at the operating stress level is considered adequate for use in missiles and unmanned space vehicles. Therefore, many of the alloys tested, incorporating these joint configurations, are acceptable for structural applications.

A decrease in the static strength of the joint, with decreasing temperature and a low number of cycles to failure, generally indicates low-temperature embrittlement. This occurs in AM-355 stainless steel at -320 and -423°F. However, poor fatigue results may be attributed to a low tension/shear ratio of the resistance spot welds. This is true for the Ti-5Al-2.5Sn alloy since butt fusion welded joints, without reinforcements and spot welds, possess much better fatigue properties. The low tension/shear ratio of the resistance spot welds for this alloy, as well as other titanium alloys, is the cause of the poor fatigue properties in complex welded joints.

Static and axial fatigue tests on complex welded joints correlate quite well with static and repeated loading tests on full size propellant tanks and stub tanks tested at room and low temperature. Therefore, considerable importance is attached to static and axial fatigue properties of complex welded joints in selecting materials for thin-walled, pressure stabilized propellant tanks.

Figures 15 through 18 present data of ultimate tensile strength, joint efficiencies, and elongation values for heliarc butwelds in stainless steels, aluminum alloys, titanium alloys, and nickel base alloys down to -423°F (Ref. 40).

Table 10. Axial Fatigue Properties of Complex Welded Joints

Alloy	Test Temp., °F	Static Strength, Psi	Stress Range, Psi	Number of Cycles to Failure
Type 301 stainless 60% CR*	78	222,000	0-150,000	862
			0-170,900	544
			0-190,000	420
	−320	259,000	0-189,000	1029
			0-214,000	406
			0-239,000	74
	−423	209,000	0-157,000	73
			0-178,000	53
			0-194,000	4
Type 304L stainless 50% CR	78	182,000	0-134,000	481
	−320	235,000	0-166,000	1349
	−423	251,000	0-196,000	512
Type 310 stainless	78	187,000	0-117,000	1251
			0-133,000	574
			0-149,000	398
	−320	259,000	0-170,000	1745
			0-193,000	690
			0-216,000	302
	−423	286,000	0-199,000	794
			0-225,000	325
			0-252,000	133
AM-355 stainless CRT†	78	290,000	0-236,000	139
	−320	148,000	0-126,000	5
	−423	99,000	0- 85,000	19
2014-T6 aluminum (butt fusion weld only)	78	43,700	0- 34,200	2462‡
			0- 38,800	1788‡
			0- 43,400	2003‡
	−320	54,800	0- 41,200	2000‡
			0- 46,700	1958‡
			0- 52,200	1338‡
	−423	71,000	0- 49,900	2000‡
			0- 56,500	1665‡
Ti-5Al-2.5Sn annealed	78	120,000	0- 87,000	1604
			0- 99,000	950
			0-110,000	440
	−320	188,000	0-140,000	315
			0-159,000	57
			0-178,000	9
	−423	172,000	0-129,000	663
			0-146,000	55
			0-163,000	11
Ti-5Al-2.5Sn annealed (butt fusion welded only)	78	121,000	0-102,000	141
	−320	192,000	0-140,000	2100‡
	−423	233,000	0-140,000	2000‡
			0-192,000	567

*CR cold rolled; †CRT=cold rolled and tempered; ‡Specimen did not fail—test was stopped.

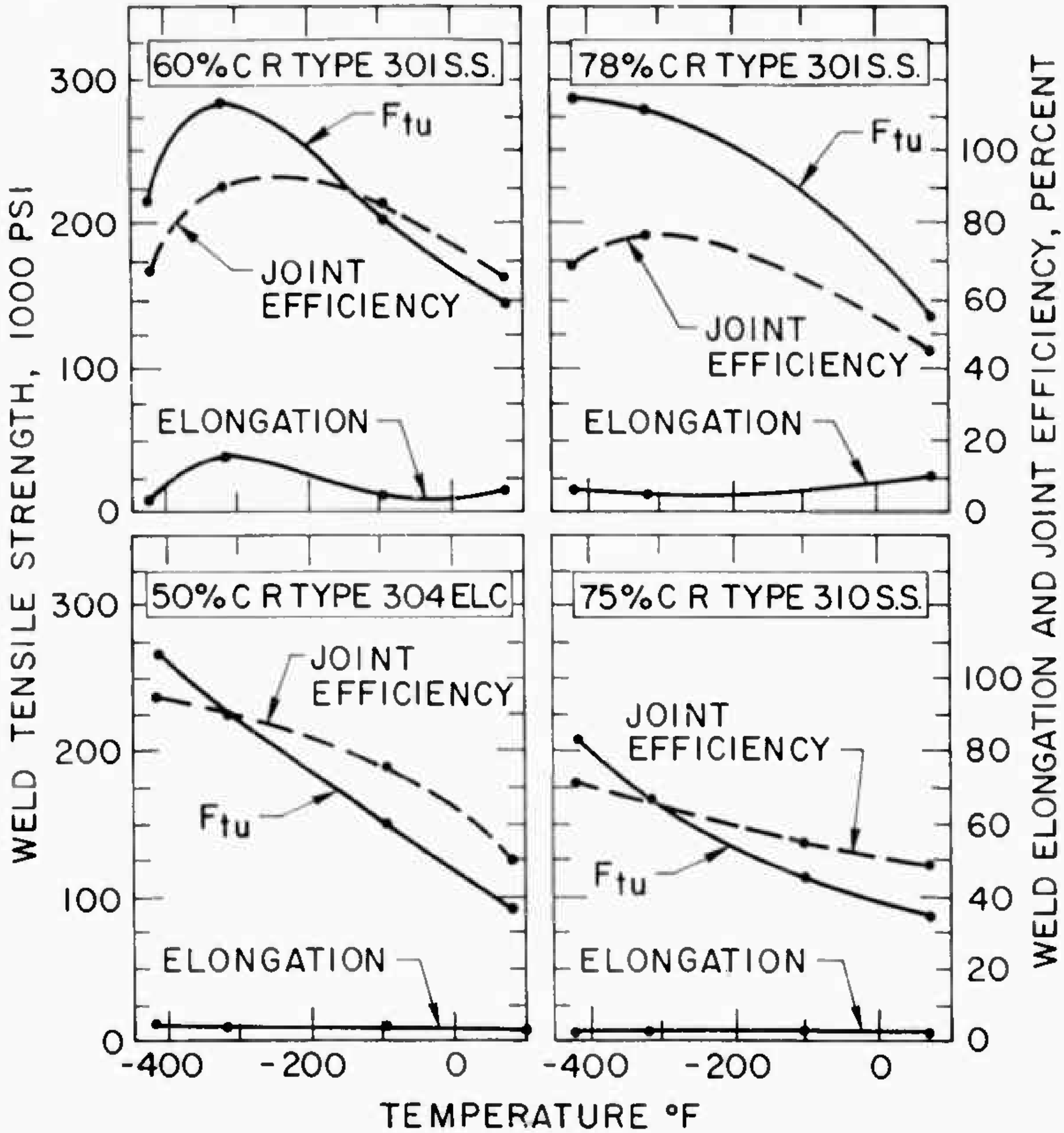

Fig. 15. Properties of Heliarc Butt Welds in Stainless Steels

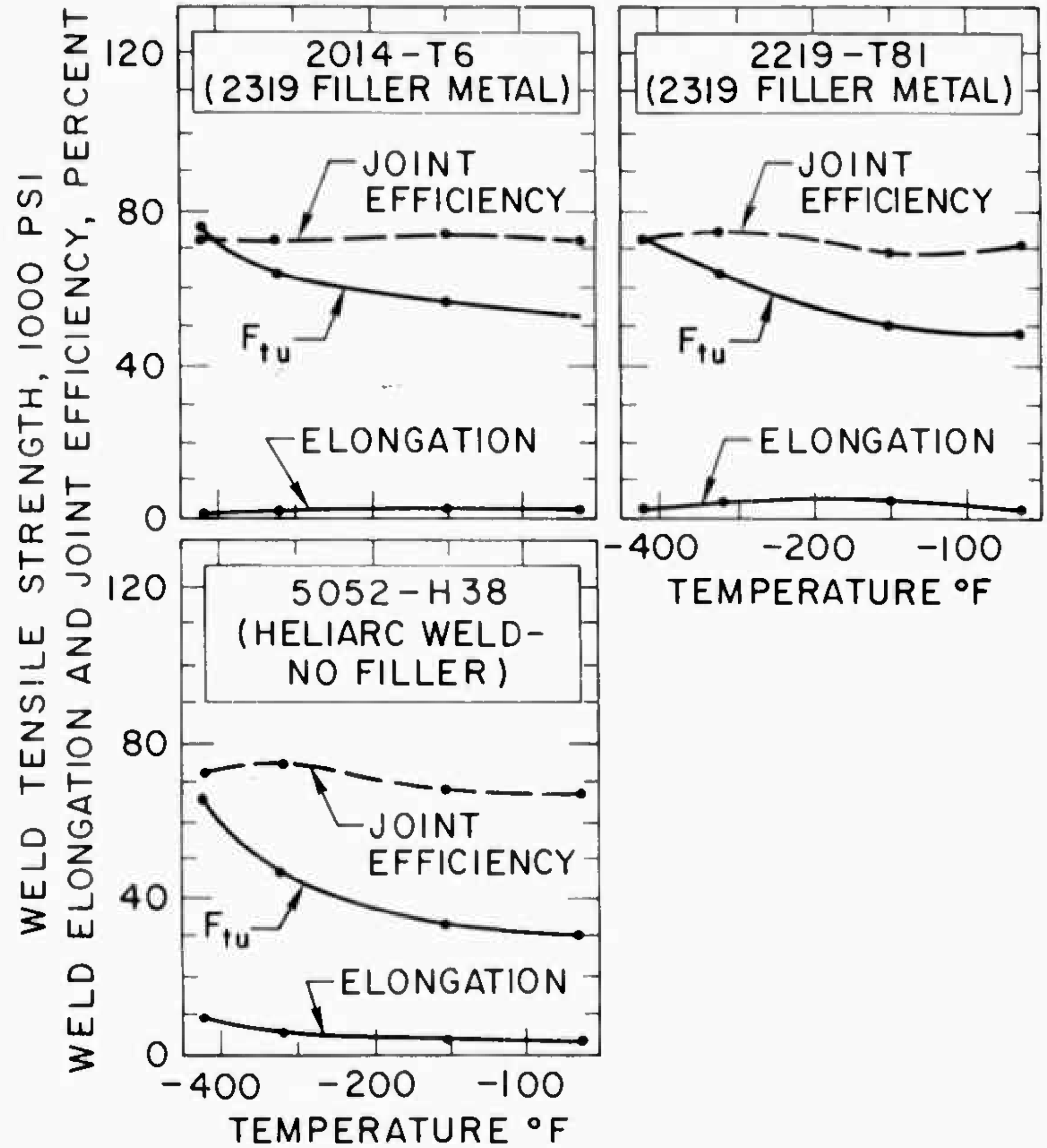

Fig. 16. Properties of Fusion Butt Welds in Aluminum Alloys

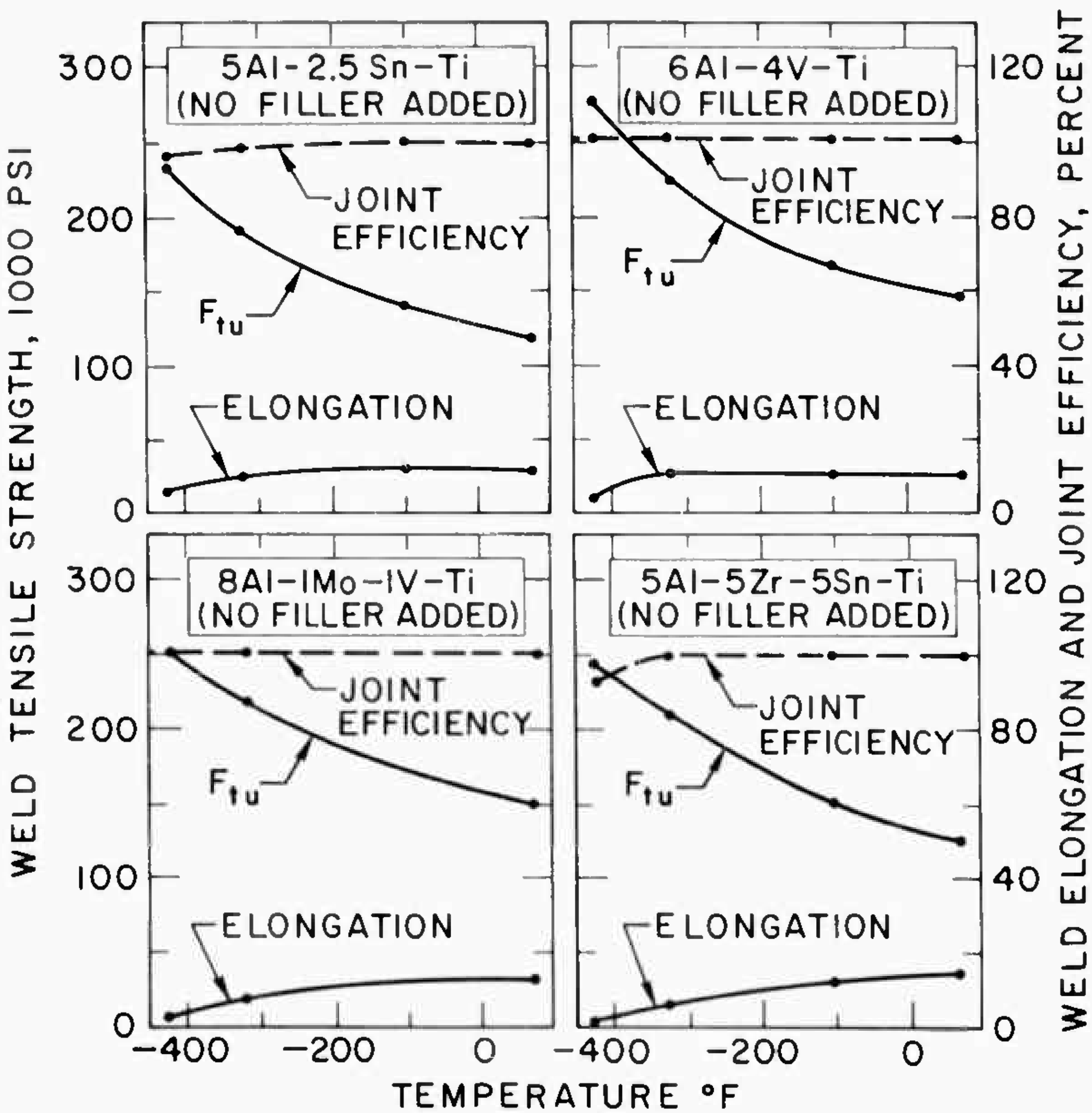

Fig. 17. Properties of Heliarc Butt Welds in Titanium Alloys

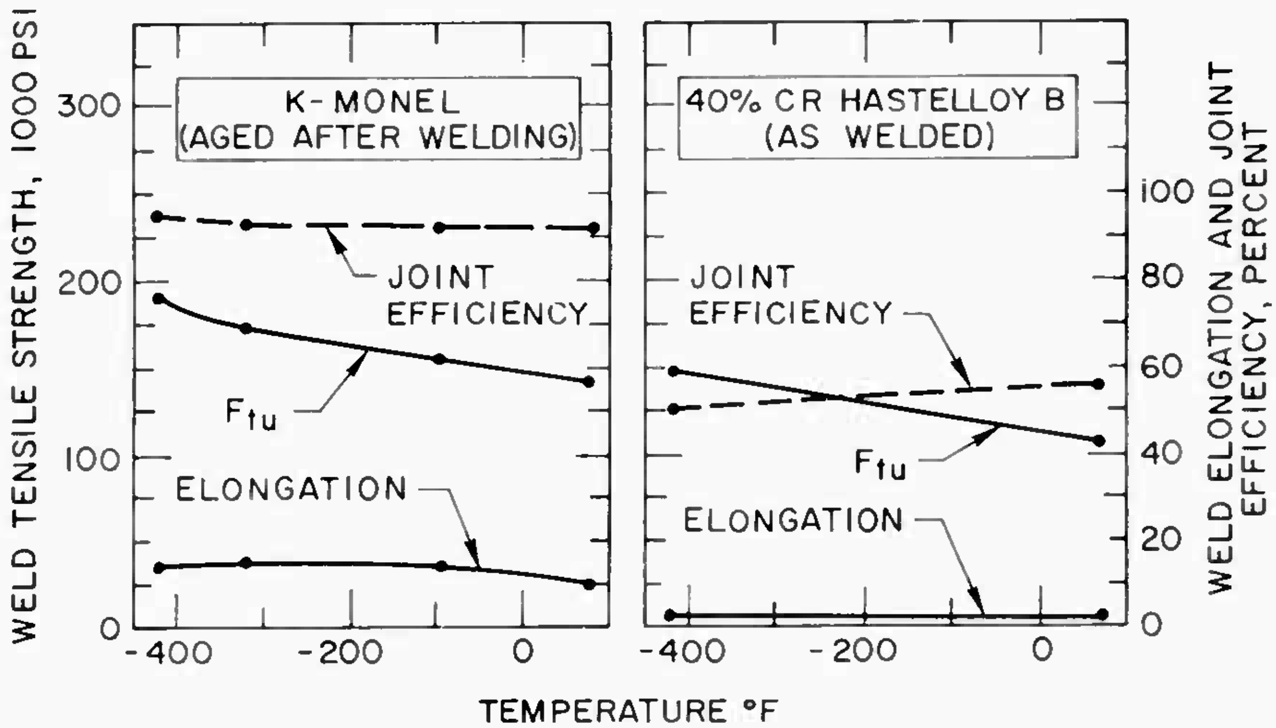

Fig. 18. Properties of Heliarc Butt Welds in Nickel-Base Alloys

XII. APPLICATIONS AND SUMMARY

Table 11 lists the alloys that have been selected for the cryogenic propellant and pressurant tankage in the various missile and booster systems. Austenitic stainless steels, aluminum alloys, and titanium alloys predominate. The trend today is in the direction of obtaining the highest strength/weight ratio material at the temperature of application. In Fig. 19, plots of the yield/and tensile strength/weight ratios with respect to temperature indicate the superiority of the Ti-5Al-2.5Sn alloy in this respect. Although 310 stainless steel was selected for the liquid hydrogen tankage on the earlier established Centaur system, in a recent recommendation (Ref. 38), the Ti-5Al-2.5Sn alloy is selected for similar use on a LOX-LH_2 fueled, recoverable aerospaceplane.

In summary, although some precipitation hardened alloys have good low-temperature properties, single-phase, solid solution strengthened alloys having close-packed atomic structures are preferred for extreme low temperatures ($\leq 423^{\circ}F$). If an invitation to brittle fracture is to be avoided, it is recommended that precipitation hardened alloys not be used. It is recognized that tantalum presents an anomaly to this concept. No explanation can be offered at this time for this behavior, however, it is believed to be somewhat dependent upon such things as stacking fault and surface energies.

Table 11. Alloys for Propellant and Pressurant Tankage

PROPELLANT	SYSTEM	ALLOY
LOX	V-2	Al-5% Mg
	JUPITER	Al 5052
	JUPITER-C	Al 5086
	THOR	2014-T6
	ATLAS	301 XFH
	TITAN I	2014-T6
	SATURN	Al 5456-H343(H321)
	CENTAUR	301 XFH
LH_2	CENTAUR	310(75% CR)
	SATURN	2014-T6
	SPACEPLANE	Ti-5Al-2.5Sn(ELI)
LN_2	ATLAS	Ti-6Al-4V

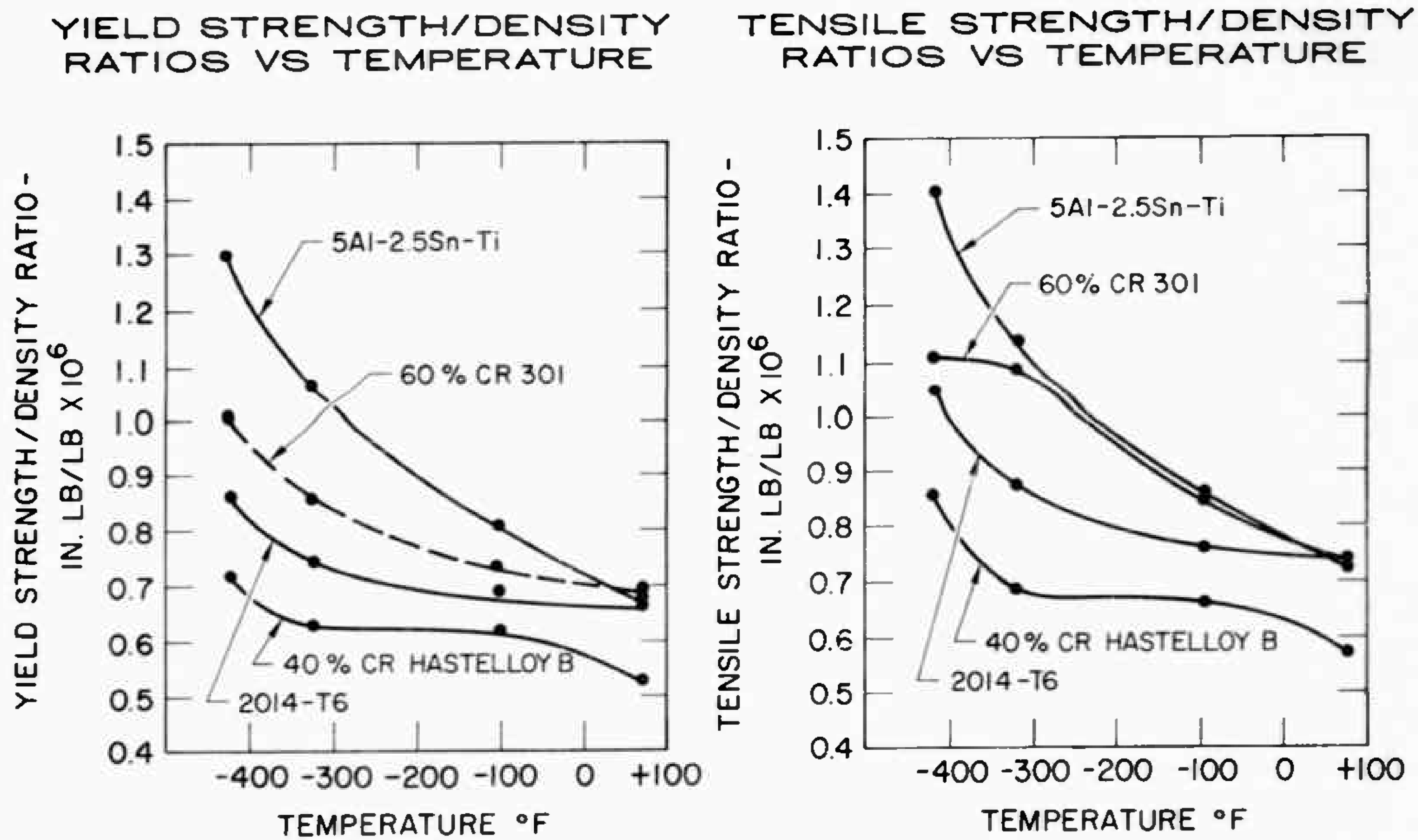

Fig. 19. Yield and Tensile Strength/Weight Ratios for Various Alloys at Various Temperatures

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