Technical

Report

Final Report
F-C3013

DEVELOPMENT OF ULTRA FINE GRAIN SIZE TITANIUM WITH IMPROVED MECHANICAL PROPERTIES

By

Robin L. Jones

March 1972

Prepared for

NAVAL AIR SYSTEMS COMMAND

Contract No. N00019-71-C0133

[redacted]

THE FRANKLIN INSTITUTE RESEARCH LABORATORIES
BENJAMIN FRANKLIN PARKWAY • PHILADELPHIA, PENNA 19103

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ABSTRACT

A two-part study of the effects of interstitial content, grain refinement and cold working on the mechanical properties of titanium-base materials was conducted. In the first part techniques were developed to refine the grain size of Ti-5Al-2.5Sn into the range of 0.5 microns, grain growth kinetics were evaluated and room temperature tensile behavior was investigated. Grain refinement leads to an increase of yield strength (of up to 60 ksi), which obeys the Hall-Petch relation, an increase in reduction in area and a decrease of uniform elongation. Cold working after recrystallization to submicron grain size results in a further strength increase (up to 30 ksi) and a substantial further elongation decrease. Apparent age hardening behavior was detected in thermal stability tests which established that the short term stability limit of the ultrafine microstructure is close to the recrystallization temperature. In the second part of the program the effects of grain size and cold work on the room temperature toughness of Grade 3 unalloyed titanium sheet were investigated. Notched tensile, modified Kahn-type tearing, and fatigue crack propagation tests were employed. Strength increases were found to correspond to improved tear strength and reduced fatigue crack propagation rates while other toughness parameters were generally raised to some extent by grain refinement and lowered by cold working.

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A two-part study of the effects of interstitial content, grain refinement and cold working on the mechanical properties of titanium-base materials was conducted. In the first part techniques were developed to refine the grain size of Ti-5Al-2.5Sn into the range of 0.5 microns, grain growth kinetics were evaluated and room temperature tensile behavior was investigated. Grain refinement leads to an increase of yield strength (of up to 60 ksi), which obeys the Hall-Petch relation, an increase in reduction in area and a decrease of uniform elongation. Cold working after recrystallization to submicron grain size results in a further strength increase (up to 30 ksi) and a substantial further elongation decrease. Apparent age hardening behavior was detected in thermal stability tests which established that the short term stability limit of the ultrafine microstructure is close to the recrystallization temperature. In the second part of the program the effects of grain size and cold work on the room temperature toughness of Grade 3 unalloyed titanium sheet were investigated. Notched tensile, modified Kahn-type tearing, and fatigue crack propagation tests were employed. Strength increases were found to correspond to improved tear strength and reduced fatigue crack propagation rates while other toughness parameters were generally raised to some extent by grain refinement and lowered by cold working.

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1. INTRODUCTION

In the course of two previous research programs supported by Naval Air Systems Command [1,2] it was established that the room temperature strength of commercial purity unalloyed titanium wire and sheet could be raised by up to a factor of two without serious loss of ductility by combining the effects of grain refinement and cold deformation. During the second program [2] it was demonstrated that improvements in fatigue behavior (particularly low cycle fatigue) and stress corrosion resistance in $FeCl_3$/methanol accompanied the strength increase. The thermal stability of the fine grain size and cold worked microstructures was investigated and it was shown that at room temperature the resistance of the strengthened materials to stress raising notches remained high.

The initial major objective of the present program was to investigate the feasibility of applying thermomechanical processing procedures similar to those developed for the grain refinement of unalloyed titanium to commercially available single phase titanium alloys in order to determine whether or not similar improvements in mechanical properties could be obtained in alloyed materials. A second major objective, introduced by the sponsor during the program, was to investigate in detail the effects of grain size refinement and cold deformation on the room temperature toughness of unalloyed titanium sheet produced by the methods developed previously. In order to accommodate this second objective the program plan was somewhat changed from that originally proposed. Studies on alloyed materials were chiefly confined to the α-alloy Ti-5Al-2.5Sn and only preparatory experiments were performed with single phase bcc alloys. The experimental program thus consisted of two distinct parts, which for the sake of clarity, are discussed separately throughout this report.

2. EXPERIMENTAL RESULTS - TITANIUM ALLOYS

2.1 Experimental Materials

One α-alloy and three β-alloys were investigated during the program. The α-alloy, Ti-5Al-2.5Sn was obtained in two purities called regular grade and ELl (extra low interstitial) grade throughout the report. All materials were obtained in the form of 1/4 inch drawn rod for wire experiments and the two grades of α-alloy were also obtained in the form of 1/2 inch plate for sheet studies. The ELI purity plate and the β-alloy rods were kindly provided by NRL and Standard Pressed Steel respectively. The other materials were obtained commercially. As-received analyses and mechanical properties are tabulated in Tables 2.1 and 2.2. All materials were received in the annealed or solution treated condition.

2.2 Mechanical Reduction Procedures

In the previous work on unalloyed titanium (1,2) it was found that the largest possible cold reduction before annealing was required to obtain the finest recrystallized grain sizes. The initial step in the alloy investigation was therefore aimed at assessing the cold reduction attainable by swaging and rolling which did not result in seriously flawed material. The effect of the cold reduction on mechanical properties was followed by Vickers hardness measurements. The correlation curve for hardness and yield strength obtained previously for unalloyed titanium is reproduced as Figure 2-1. Data for the α-alloys were found to agree with this correlation while β-alloys were found to exhibit lower hardness values for a given yield strength than α-alloys or unalloyed material. The effect of swaging reduction on hardness for Ti-5Al-2.5Sn of two purities is shown in Table 2.3. As observed for unalloyed material the hardness rises continuously with reduction indicating the necessity for a large initial reduction if the finest grain sizes are to be obtained.

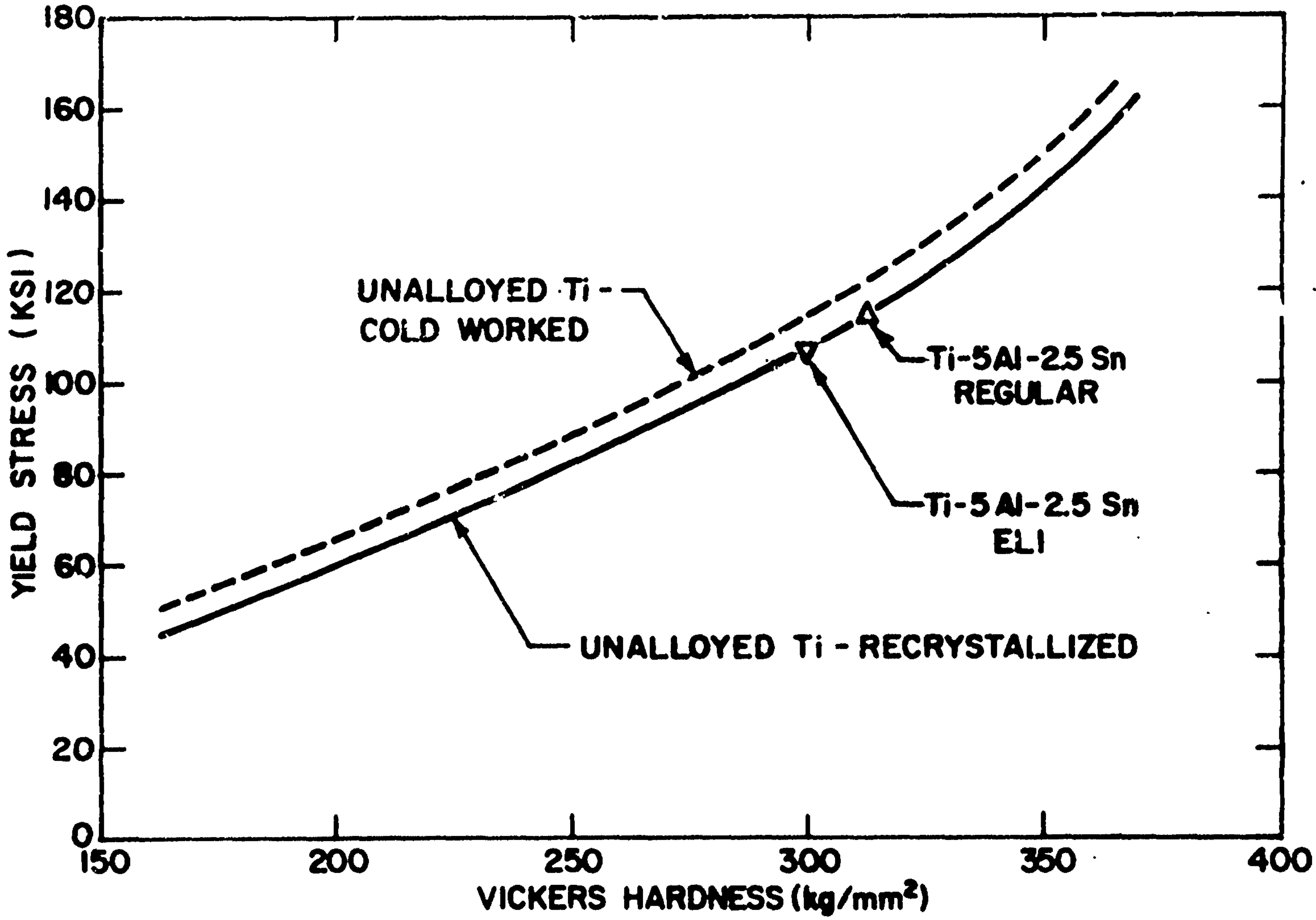


Fig. 2-1. Correlation Curves of Yield Stress and Vickers Hardness Developed for Unalloyed Titanium Have Been Found to Apply to Ti-5Al-2.5Sn but Not to the β-Alloys Investigated.

TABLE 2-1

MANUFACTURERS ANALYSES FOR THE EXPERIMENTAL MATERIAL

1. Ti-5Aℓ-2.5 Sn Regular Grade Rod and Plate

Aℓ	Sn	Fe	O	C	N	H	Ti	
5.1	2.7	0.4	0.18	0.05	0.02	<0.01	Balance	wt%

2. Ti-5Aℓ-2.5Sn ELI Grade Rod

Aℓ	Sn	Fe	O	C	N	H	Ti	
5.2	2.6	0.09	0.1	0.013	0.014	<0.006	Balance	wt%

3. Ti-5Aℓ-2.5 Sn ELI Grade Plate

Aℓ	Sn	Fe	O	C	N	H	Ti	
5.2	2.5	0.16	0.07	0.025	0.014	0.004	Balance	wt%

4. RMI Beta C (38644) Rod

Aℓ	V	G	Mo	Zr	Fe	O	C	N	H	Ti	
3.4	7.8	5.8	4.1	3.7	0.16	0.09	0.02	0.01	21 ppm	Bal.	wt%

5. Crucible Beta III Rod

Mo	Zr	Sn	Fe	O	C	N	H	Ti	
11.6	6.3	4.4	0.04	0.1	0.04	0.01	0.004	Bal.	wt%

6. TMCA Ti 8823 Rod No analysis supplied: Typically

Mo	V	Fe	Aℓ	O	C	N	H	Ti	
8	8	2	3	0.1	0.04	0.01	0.004	Bal.	wt%

TABLE 2-2

AS-RECEIVED MECHANICAL PROPERTIES OF THE EXPERIMENTAL MATERIALS

Material	Form	0.2% Yield	Tensile	Elong.	R.A.
		ksi	ksi	%	%
5-2½ Regular	Rod/Plate	116	123	12	28
5-2½ ELI	Rod	107	120	13.5	33
5-2½ ELI	Plate	114	124	19	41
RMI Beta C	Rod	138	142	15	53
Crucible Beta III	Rod	130	142	28	70
TMCA Ti 8823	Rod	133	137	22	69

TABLE 2.3

EFFECT OF COLD REDUCTION ON THE HARDNESS OF Ti-SAl-2.5 Sn OF TWO PURITES

Material	Reduction %	Vickers Hardness (kg/mm^2)
Regular Grade	0	312
"	31	345
"	61	353
"	73	374
"	87	381
"	90	409
ELI Grade	0	300
"	51	341
"	67	345
"	79	350
"	92	368

The rate of hardness increase is more rapid for the higher interstitial content material. The α-alloy wires were found to be more difficult to swage than unalloyed material. In order to produce consistently sound wires very careful lubrication (using paraffian wax as before) was necessary and intermediate stress relief anneals were found to be advantageous. The final swaging procedure which proved to be most successful was as follows: cold swage to 50% reduction; stress relieve @ 400°C 1 hr; cold swage to overall 75% reduction; stress relieve @ 400°C 1 hr; cold swage to final reduction. The stress relieving treatments were found to have little or no effect on the observed hardness after swaging but prevented premature failure.

The three β-alloys were all substantially more difficult to cold swage than the α-alloys showing a marked tendency towards galling to the swaging die, sometimes with quite spectacular pyrophoric results. Sound material could only be obtained with the strictest attention to lubrication and with slow feed rates into the swager. The same stress relief procedures were employed as those detailed above and these allowed reduction to 90% to be attained for all 3 alloys. The effect of swaging on the Vickers hardness was somewhat curious; although the hardness increased with reduction as before the rate of increase was slow compared with that observed for the α-alloys, particularly for the β-111 material. Table 2.4 shows the initial and final hardnesses before and after 90% cold reduction. On the basis of these data it appears that the strength of β-C in the solution treated condition might be increased by thermomechanical processing by up to 70 ksi while the maximum increase for the other two alloys is probably about 40 ksi. In order to allow the inclusion in the program of the toughness studies discussed in Section 3, work on the β-alloys was terminated at this point. The questions left unanswered are; what effect does grain refinement have on the ductility and toughness of the β-alloys; why does the relation between reduction and hardness differ so markedly from that of unalloyed material and the α-alloys; why does β-C differ from the other β-alloys (possibly a stress-induced precipitation reaction or transformation); is the strengthening contribution due to grain refinement in the solution annealed condition maintained after aging and does a fine grain structure or heavily cold worked substructure modify the ageing characteristics? It is hoped that these questions can be investigated during future programs.

TABLE 2.4

THE EFFECT OF 90% COLD REDUCTION ON THE HARDNESS OF THREE β-TITANIUM ALLOYS IN THE SOLUTION TREATED CONDITION

Material	Initial Hardness (Vickers)	Final Hardness (Vickers)
RMI Beta C	278	370
Crucible Beta III	267	330
TMCA Ti 8823	271	351

Attempts to fabricate 90% cold worked Ti-5Al-2.5 Sn sheet by room temperature rolling have not proved to be very successful. Workpiece failure by 45° shear cracking is always observed before the desired reduction is attained. Severe edge cracking is observed for unidirectionally rolled plate at ∿50% reduction for regular grade material and ∿65% reduction for ELI purity material. A number of rolling procedure variations have been investigated, including use of workpiece encapsulation (to maximize the hydrostatic constraint of the workpiece), the use of intermediate annealing treatments at a range of temperatures and following various reductions, and initial warm breakdown followed by cold rolling. The most successful procedure developed consisted of 40% cold rolling increments with intermediate annealing treatments at 600°C for 1 hour. This is somewhat more than a stress relieving treatment (see Section 2-3) and leads to a detectable hardness decrease. Using this procedure reductions of ∿75% were attained before workpiece failure. The Vickers hardnesses at this reduction were ∿360 and 350 VPN for the regular and ELI purity materials respectively. These values are similar to those produced by swaging to ∿60% reduction (see Table 2.3). It is believed from previous experience that this amount of cold reduction would allow a minimum grain size of ~ 1 micron to be attained using an optimum heat treatment. It appears that to attain finer grain sizes than this in alloy sheet materials a completely different approach may be needed. Two alternative routes appear feasible in concept; powder metallurgical fabrication using prealloyed powders, or grain refinement by dynamic recrystallization during working near the recrystallization temperature. Abrahamson[3] has obtained submicron grain sizes in lightly alloyed titanium by the latter route. It is intended to pursue these possibilities during the program presently beginning. No attempt has been made to evaluate alloy sheet produced during the present program since wider variations in the variables of interest were attainable in wire samples, and the results of the previous program[1,2] indicate that sheet properties are likely to closely resemble those observed in wires.

2.3 Recrystallization and Grain Coarsening Behavior

The recrystallization and grain growth characteristics of Ti-5Al-2.5Sn of two purities have been investigated for wire samples initially cold swaged to 90% total reduction with intermediate stress relief anneals. Figure 2-2 shows the variation in Vickers hardness produced by both short and long term isothermal anneals. The 20 second annealing treatments were carried out by immersion in molten lead while the 1 hour annealing treatments were performed in a resistance heated vacuum furnace at a pressure of $\sim 10^{-6}$ torr. The surprising observation is that both materials show hardness increases for 1 hour anneals at temperatures near 500°C. This observation was subsequently investigated further and is thought to be due to a previously unreported precipitation reaction. The hardness increase is not apparent for short term anneals, probably because the precipitation kinetics are too slow to allow significant decomposition to occur in the time available. X-ray back reflection indicated complete recrystallization in both materials annealed for 1 hour at 600°C. For 20 second anneals complete recrystallization was observed at 725°C and 775°C for ELI and regular purity material respectively. Grain sizes following these latter treatments were determined by thin

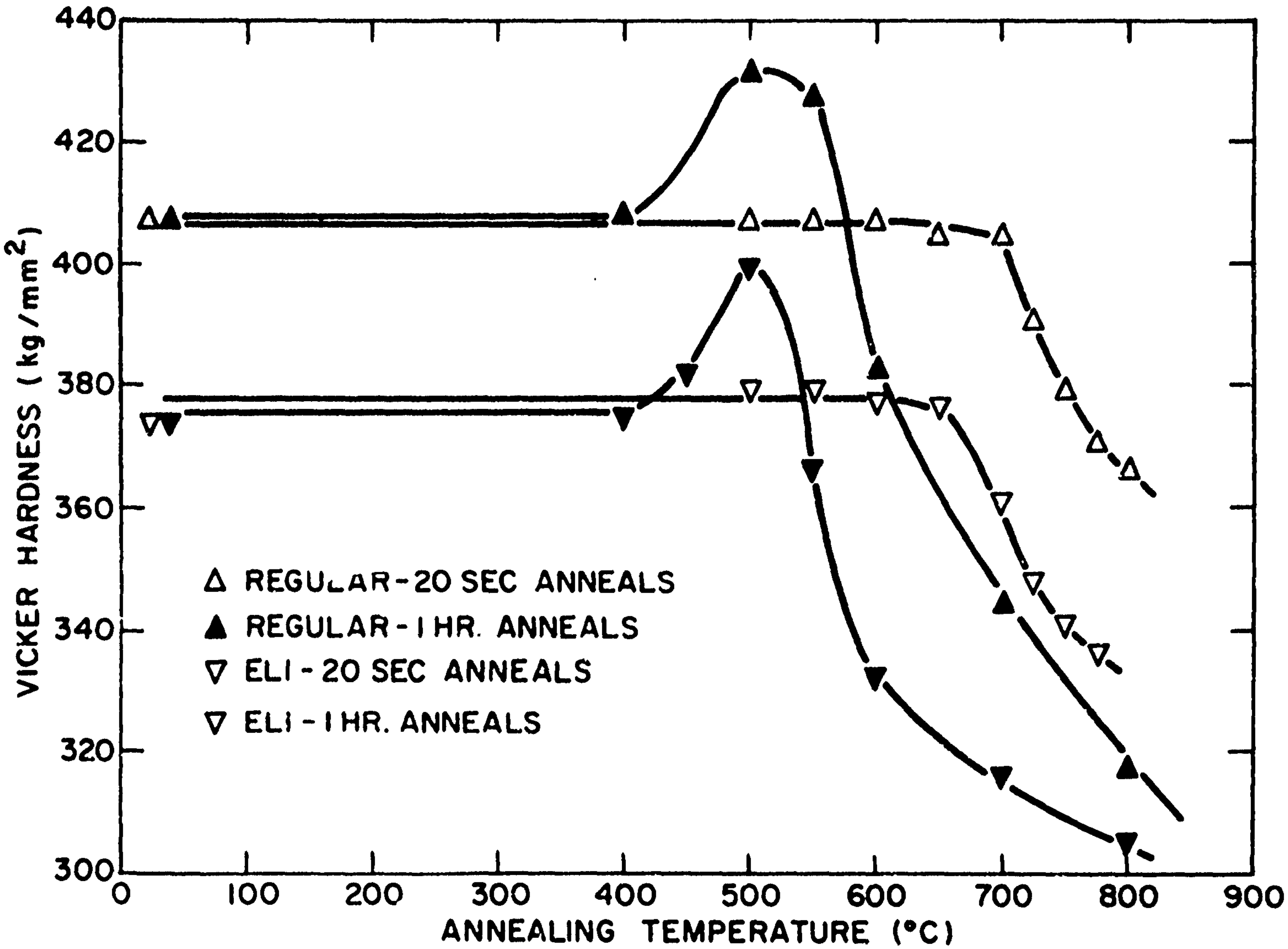


Fig. 2-2. Variation in Vickers Hardness Produced by Short and Long Term Isothermal Annealing of Initially 90% Cold Swaged Ti-5Al-2.5Sn Alloys Wires

foil transmission microscopy to be 0.5 microns and 0.4 microns for the ELI and regular purity grades respectively. No indications of the presence of second phase particles were observed in these thin foils.

The kinetics of grain growth for both regular and ELI purity material were investigated by determining grain sizes as a function of annealing time at 700°C, 800°C and 900°C. Anneals were performed in a dynamic vacuum of $\sim 10^{-6}$ torr and the grain size was equated with the mean linear intercept throughout. Grain sizes below 5 microns were determined by transmission microscopy. Larger grain sizes were measured optically. Figure 2-3 is a log-log plot of the grain size data obtained as a function of annealing time which indicates a parabolic dependence between grain size and annealing time. The grain size squared is accordingly plotted against annealing time in Figure 2-4. Growth rates for ELI are more rapid than those for regular purity material at all temperatures in accordance with the effect of interstitials on grain growth rate observed for unalloyed titanium[1,2,4]. A curious observation noted during this study was the appearance of surface cases up to 0.01 in. in thickness on samples annealed at 900°C, which showed different etching characteristics from those of the interior. Microhardness measurements indicated that the cases were harder than the interiors of the samples by up to a factor of 2 suggesting that interstitial (probably oxygen) pickup from the dynamic vacumm had occurred. The curious aspect of the observation was that the grain size in the presumably high interstitial case was larger than that in the sample interior by up to a factor of about 2. It thus appears possible that the retardation of grain growth kinetics which we normally associate with increasing interstitial content only occurs at the relatively low contents typical of commercial purity materials. At higher contents interstitials may cause accelerated grain growth. The grain sizes in the center of these samples are those plotted in Figures 2-3 and 2-4.

2.4 Effect of Grain Size and PRR on the Room Temperature Tensile Properties

Recrystallized wire samples with grain sizes in the range 0.4 microns to 10 microns were prepared for both ELI and regular purity

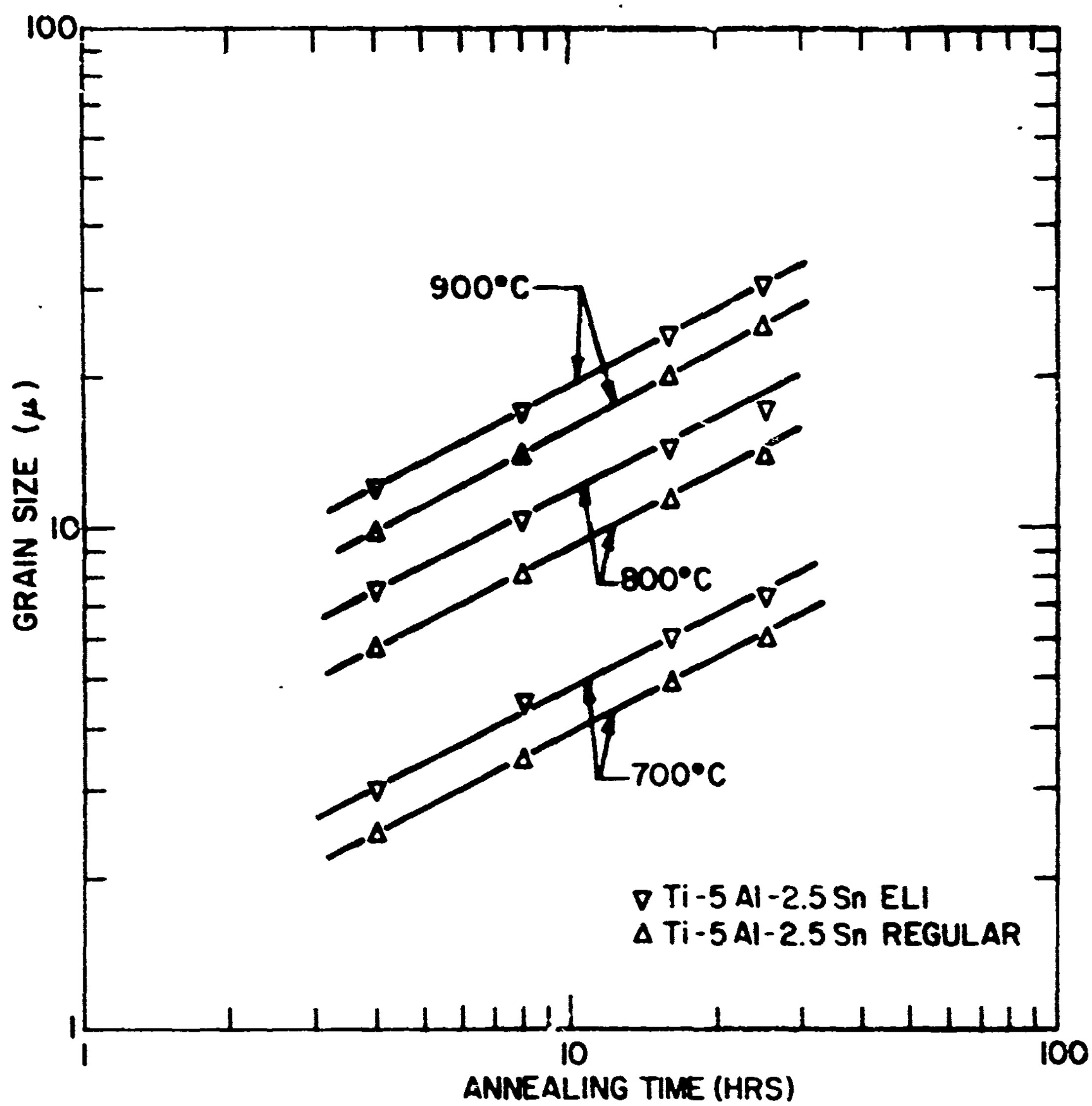


Fig. 2-3. Relation Between Grain Size and Annealing Time at Three Temperatures for Ti-5Al-2.5Sn of Two Purities

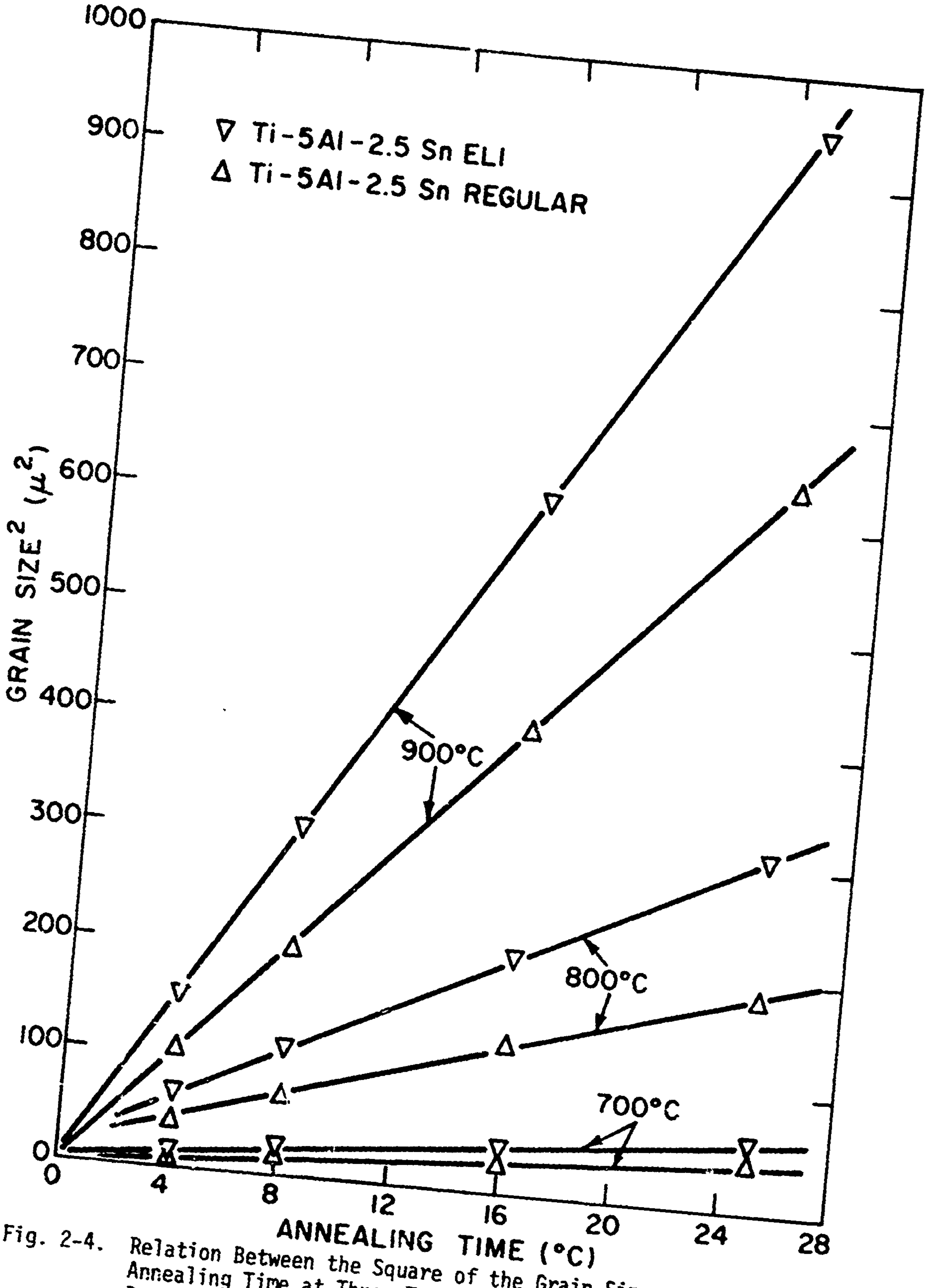


Fig. 2-4. Relation Between the Square of the Grain Size and the Annealing Time at Three Temperatures for Regular and ELI Purity Ti-5Al-2.5Sn.

material by ~90% cold swaging (with intermediate stress relief annealing) followed by isothermal annealing in the temperature range 700-800°C. Grain sizes less than about 2 microns were produced by short anneals in molten lead while longer anneals in the vacuum furnace were used for the larger grain sizes. Chemical polishing was used to produce a 1/2 inch long reduced gage section on the 0.078 inch diameter wires and then the diameter of the entire sample was further reduced by chemical polishing to facilitate testing. The final samples had ~0.06 in. diameter shoulders and ~0.05 in. diameter gage sections. Tensile tests were conducted at room temperature at a nominal strain rate of 3×10^{-4} sec^{-1}. Duplicate tests were run for all grain sizes and tensile data averaged. While yield strengths were found to be quite reproducible, flow properties, particularly elongations, were not very consistent for fine grain size samples. This appears to result from slight nonuniformities in the gage diameter which are almost inevitably produced by the chemical polishing procedure. Accordingly, additional samples with the finest grain sizes were fabricated and tested so as to obtain reasonably accurate estimates of the elongation and UTS. Table 2.5 contains the average data obtained for 0.4 micron regular grade and 0.5 micron ELI grade wires. Data for 10 micron ELI and 8 micron regular grade are included for comparison. The table also contains data for 40% PRR samples discussed later. Figure 2-5 shows all the data for the 0.2% proof stress for both materials plotted against the inverse square root of the grain size.

Wire samples for PRR studies were initially swaged 84% (with intermediate stress relief anneals) and heat treated for 20 seconds at 725°C (ELI) and 775°C (Regular). The effect of additional swaging reduction was initially monitored by hardness measurements. As noted previously for unalloyed material, hardness increased quite rapidly with reduction up to about 30-40% followed by a slow rise thereafter. 40% reduction was therefore selected as the PRR reduction for tensile investigation and test samples were fabricated for material in this condition and tested in the manner described above. Ten samples were tested for each material and the average tensile data obtained are recorded in Table 2.5.

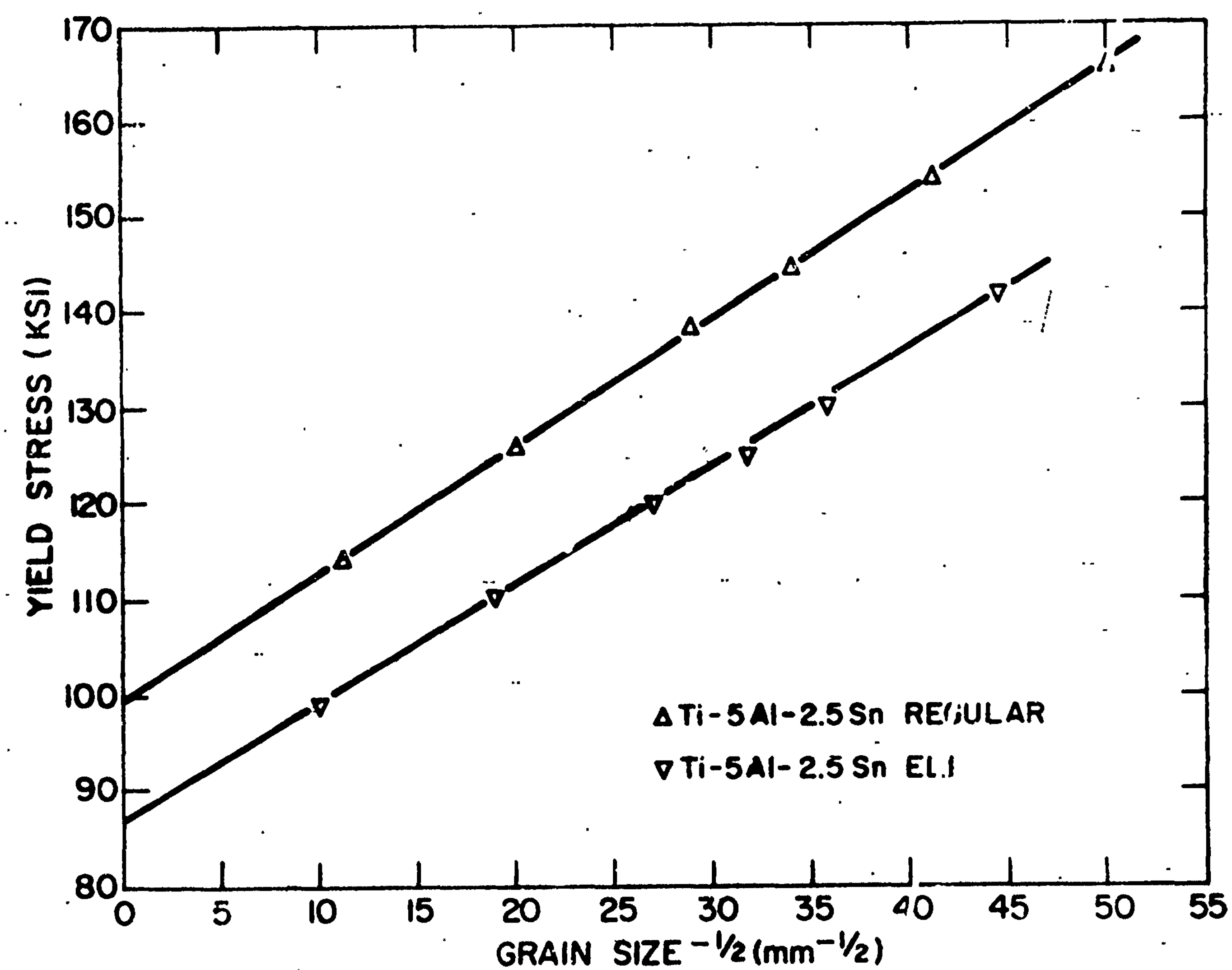


Fig. 2-5. Relation Between the 0.2% Offset Yield Stress and the Inverse Square Root of the Grain Size for Regular and ELI Purity Ti-5Al-2.5Sn Alloys.

TABLE 2.5

ROOM TEMPERATURE TENSILE DATA FOR Ti-5Aℓ-2.5Sn ALLOY WIRES

Material	Grain Size	σ_y	σ_u	e_u	e_{tot}	R.A.
	(micron)	(ksi)	(ksi)	(%)	(%)	(%)
Regular Grade	8	114	140	8	12	54
"	0.4	166	174	4	8	70
"	40% PRR	193	197	1.5	5	67
ELI Grade	10	99	133	16	21	55
"	0.5	142	151	9	15	62
"	40% PRR	179	188	3	10	55

2.5 Thermal Stability

The thermal stability of the ultrafine microstructure to isothermal annealing has been investigated by measuring the change in room temperature hardness resulting from 1 hour anneals at a range of temperatures. Since the PRR microstructure was expected to be the least stable to thermal effects the initial investigation was restricted to this condition. The Vickers hardness data obtained are recorded in Table 2.6.

TABLE 2.6

ROOM TEMPERATURE HARDNESS RESULTING FROM 1 HOUR ANNEALS FOR Ti-5Al-2.5Sn WIRES IN THE 40% PRR CONDITION

Regular Grade		ELI Grade	
Annealing Temperature °C	Vickers Hardness	Annealing Temperature °C	Vickers Hardness
RT	390	RT	360
500	431	500	400
550	427	550	366
600	381	600	332
700	351	700	316
800	371	800	305

As noted in the recrystallization studies, a hardness increase is observed on annealing at 500°C for both materials. At 600°C, the recrystallization temperature determined in the recrystallization study (Section 2.3), both materials show hardnesses below the initial value and further hardness decreases are observed at higher temperatures.

The hardness increase on annealing at 500°C was briefly investigated further using 90% cold swaged EL1 material. The effect of annealing time at 500°C on the room temperature hardness was measured for this material and the resulting data are reported in Table 2.7, and show a hardness peak corresponding to an annealing time of 30 minutes.

TABLE 2.7

ROOM TEMPERATURE HARDNESS RESULTING FROM ISOTHERMAL ANNEALING AT 500°C OF 90% COLD SWAGED EL1 MATERIAL

Annealing Time (Min.)	Vickers Hardness (Kg/mm^2)
0	360
15	375
30	400
60	382
120	370

3. EXPERIMENTAL RESULTS - UNALLOYED TITANIUM SHEET

3.1 Experimental Material

The material chosen for the sheet toughness study was that referred to previously as Grade 3(1,2). It conforms to ASTM Spec. #B265-58T Grade 3 and the as-received analysis is given below.

TABLE 3.1

AS-RECEIVED ANALYSIS OF GRADE 3 BILLET

O(ppm)	C(ppm)	N(ppm)	H(ppm)	Fe(ppm)
1200	200	50	< 100	1100

Recrystallized sheet samples with grain sizes in the range 0.5 - 20 microns were produced by an initial cold reduction of 90%, without intermediate annealing, followed by isothermal annealing in the range 550°C - 700°C. Grain sizes less than about 2 microns were produced by short annealing treatments in the molten lead bath while the longer anneals required for coarser grain sizes were performed in a resistance heated vacuum annealing furnace. Test samples were prepared before annealing, and following heat treatment were surface ground to a 600 grit final finish. The grinding direction was parallel to the tensile axis of the sample which in the present series of tests was parallel to the rolling direction. Grain sizes were identified with mean linear intercepts measured by one of three procedures. Fine grain sizes ($\leq$ 1 microns) were measured by transmission electron microscopy of thin foils. Coarse grain sizes (> 5 microns) were determined by optical microscopy while for intermediate grain sizes a novel technique was developed. This consisted of fracturing a sample by 3 point loading in 0.1N $FeCl_3$/methanol and examining the fracture surface by SEM. Since the stress corrosion cracking is entirely intergranular in nature this is a convenient method of revealing both the grain shape and size. A typical fractograph is shown in Figure 3-1.

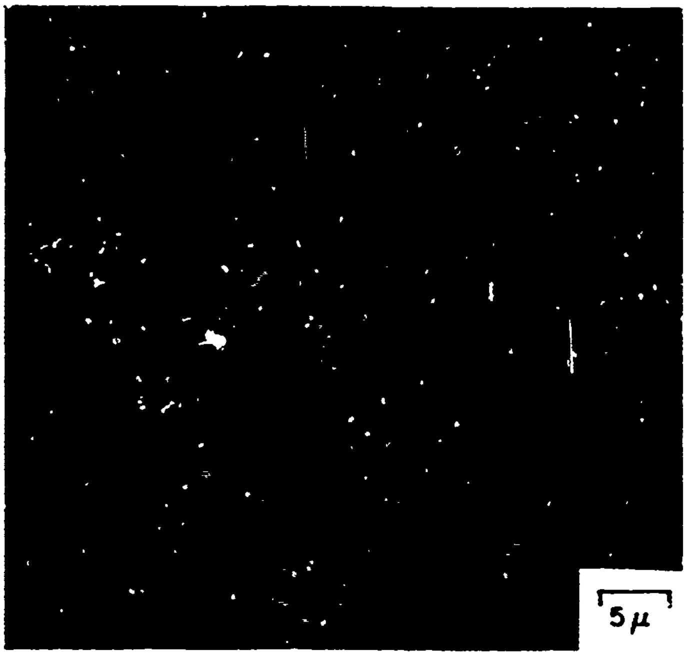


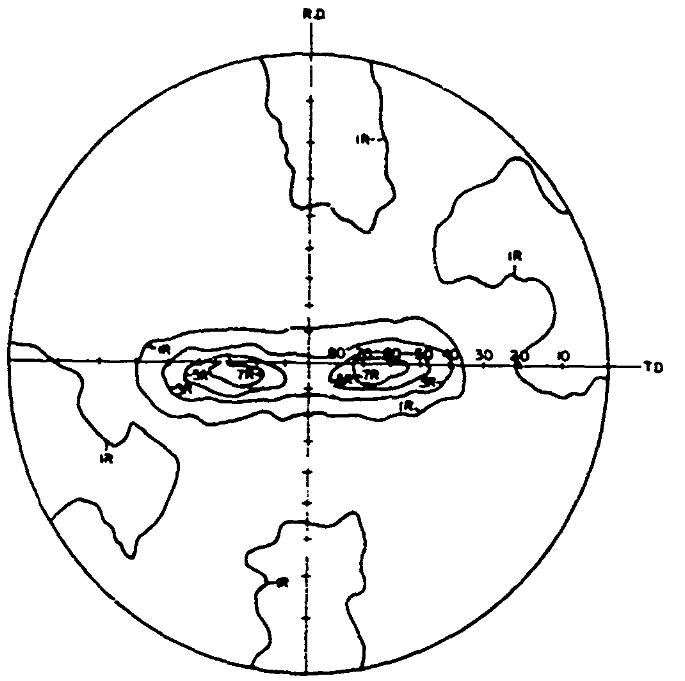
Fig. 3-1. SEM Fractograph of 5 Micron Grain Size Grade 3 Sheet, Broken by SCC in $FeCl_3$/Methanol and Used for Grain Size Determinations at Intermediate Grain Sizes.

Yield strengths were monitored using room temperature hardness measurements in conjunction with the hardness/yield strength relation developed previously[1,2]. The fabrication procedure resulted in very similar crystallographic textures for all recrystallized samples. A typical example of a basal texture is shown as Figure 3-2a.

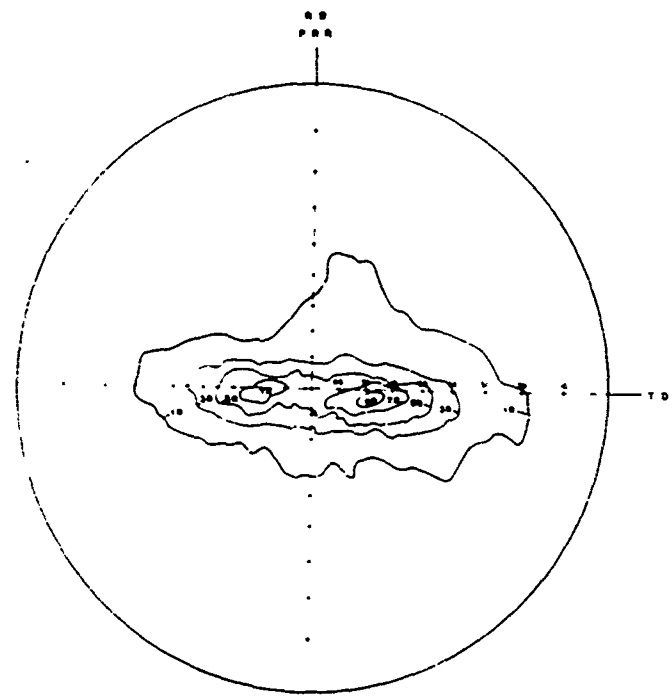
In order to obtain worked sheet with the same thickness as the recrystallized sheet (∿ 1/16 in.) a slightly different fabrication procedure was used from that employed previously[2]. Grade 3 plate was cold worked ∿ 75%, recrystallized to a grain size of ∿ 1 micron, then subjected to a 50% post recrystallization reduction (PRR) so as to obtain 1/16 in. thick sheet. The rolling direction for PRR was parallel to the rolling direction for the pre-recrystallization cold reduction and this led to the development of a very similar crystallographic texture to that of the rolled and recrystallized material (Fig. 3-2b). Two recovery treatments were used to provide samples with lower levels of stored energy, namely 20 sec @ 500°C; 5 min @ 500°C. Yield strengths for samples in the three worked conditions were determined by tensile testing at room temperature. Values obtained were 100 ksi, 110 ksi and 115 ksi for the 5 min @ 500°C, 20 sec @ 500°C and as-worked conditions respectively with the tensile axis parallel to the rolling direction. The value for the as-worked samples is about 10 ksi less than that reported previously[2] probably reflecting the somewhat larger intermediate grain size obtained by the present fabrication procedure. (The previous fabrication procedure was cold work 90%, recrystallize to 0.5 microns + 50% PRR. It was not possible to duplicate this in the present instance because of the desire to obtain a final sheet thickness of 1/16 in.)

3.2 Toughness Testing Procedures

It is important to know whether or not the increases in strength which accompany grain refinement and cold work in α-titanium are accompanied by changes in the toughness of the material. The most general definition of toughness is the energy required to break a material and thus in the simplest case the area under the tensile stress-strain curve



(a) 0.5 Micron Recrystallized Sheet



(b) 50% PRR Sheet

Fig. 3-2. Basal Textures for Grade 3 Sheet Samples gives a measure of the toughness. However, in many materials the presence of a crack causes a low-stress failure. That is to say, the failure stress is reduced to a greater extent than would be predicted by simply considering the reduction of cross-sectional area due to the cracks. In such a material a more meaningful definition of toughness is the resistance of the material to crack propagation since this determines the strength that the material will exhibit in the presence of a crack. This definition is the basis of the approach known as fracture toughness. The extensive analytical and experimental work in this area since the late 1950's has lead to the emergence of a parameter, K_{Ic}, which is considered to be a true material constant expressing the resistance of a material to crack propagation. K_{Ic}, the plane-strain fracture toughness, is defined as the critical value of the stress intensity factor K_I at the point of instability of crack extension. The subscript I refers to the opening (plane strain) mode of crack extension.

K_{Ic} is of particular importance because it represents a practical lower limit to the fracture toughness of a material under given conditions. In the present instance it is impossible to directly measure the variation of K_{Ic} with the parameters of interest (grain size and cold work) and further, it may be argued that the plane strain fracture toughness of the present materials is of no direct significance, since they will never fail in the crack opening mode. To see this let us consider the requirements for obtaining predominantly plane-strain failure.

The concept of K_{Ic} due to Irwin et al[5] entails two independent size effects associated with the two theoretically essential conditions of linear elastic behavior of the material over a field which is large compared with the plastic enclave that surrounds the crack front, and tritensile plane strain constraint within this enclave and somewhat beyond it. Any useful K_{Ic} test method must provide for these basic limitations by restriction of the valid range of a test to that for which the plastic enclave size is within some specified fraction of the most critical dimension of the specimen. In ASTM Method E 399-70T for example the plastic enclave factor R, defined as

$$R = \left(\frac{K_{Ic}}{\sigma_y}\right)^2 \quad \text{where } \sigma_y \text{ is the 0.2\% proof stress} \tag{3-1}$$

is restricted to the smaller of 0.4 a_o or 0.4B where a_o is the initial crack size and B is the specimen thickness. In the present case B $\gtrsim$ 0.06 in., hence the maximum value of K_{Ic} which can be determined for the present material (at a yield stress level of 100 ksi) is $\sim$ 15.5 ksi $\sqrt{\text{in.}}$ For commercial purity titanium of conventional grain size a typical value of K_{Ic} is $\geq$ 80 ksi $\sqrt{\text{in.}}$ Thus unless a truly catastrophic reduction in K_{Ic} accompanies grain refinement or cold work (which is extremely unlikely) it is impossible to measure values for K_{Ic} for the present sheet materials. Furthermore the fabrication techniques used to prepare these sheet materials cannot be utilized at much larger thicknesses (for instance, the flash heat treatments necessary for obtaining the finest grain sizes are impractical at thicknesses greater than about 0.2 in. because of thermal conductivity limitations) so that plane strain failures cannot be expected to occur in service.

From this discussion it is evident that we must measure toughness parameters other than K_{Ic} if we wish to characterize the effects of grain size and cold work on the toughness of unalloyed titanium sheet. Since no other toughness parameters have the material constant characteristic of K_{Ic} it seems most appropriate to investigate the effects of the material variables on a range of toughness parameters corresponding to various situations which the materials might encounter in service. Three distinct types of test have been employed and are discussed below.

3.2.1 The Notched Tensile Test

This test, which explores the notch resistance of the material under simple uniaxial tensile loading was used during the previous program[(2)]. The tensile properties are measured for symmetrical edge-notched sheet samples as a function of the stress concentration factor of the notch k_t defined as

$$k_t = 1 + 2\sqrt{a/r} \qquad (3-2)$$

where a is the half length of the notch and r is the root radius Two parameters of interest can be deduced from this test. The notch strength ratio (NSR) defined as notched UTS/unnotched UTS is a measure of the notch sensitivity of the material. Since this parameter does not depend in any way on the absolute stress level its most obvious utility as a toughness indicator occurs when comparing materials of equal or similar strengths. A second parameter which can be deduced from the test which we shall call the unit fracture energy (UFE) is defined as the area under the load-elongation curve divided by the cross section of the sample at the location of the notch. This parameter contains the stress and hence can be used to compare the toughnesses of materials of different strengths in the presence of a notch of constant stress concentration. The parameter must be used with care since it is expected to be sensitive to specimen geometry and size but with this provision it should be a valid indicator of the effect of material variables of the type of interest here.

No new experiments have been performed during this contract. However, the data obtained previously for Grade 3 sheet in three thermomechanical conditions tested longitudinally are reproduced in Table 3.2. Values of UFE for the most severely notched samples have been obtained by measuring the load-elongation areas with a planimeter and are included in the table.

3.2.2 The Tear Test

This test, which is based on the approach to Kahn and Imbembo[6] is used to investigate the propagation of a notch under the combined effects of tensile and bending stresses. The sample used, shown in Figure 3-3a, is a single edge notched sample which is pin loaded in tension. The notch root lies on the line joining the axes of the loading pins so that at a

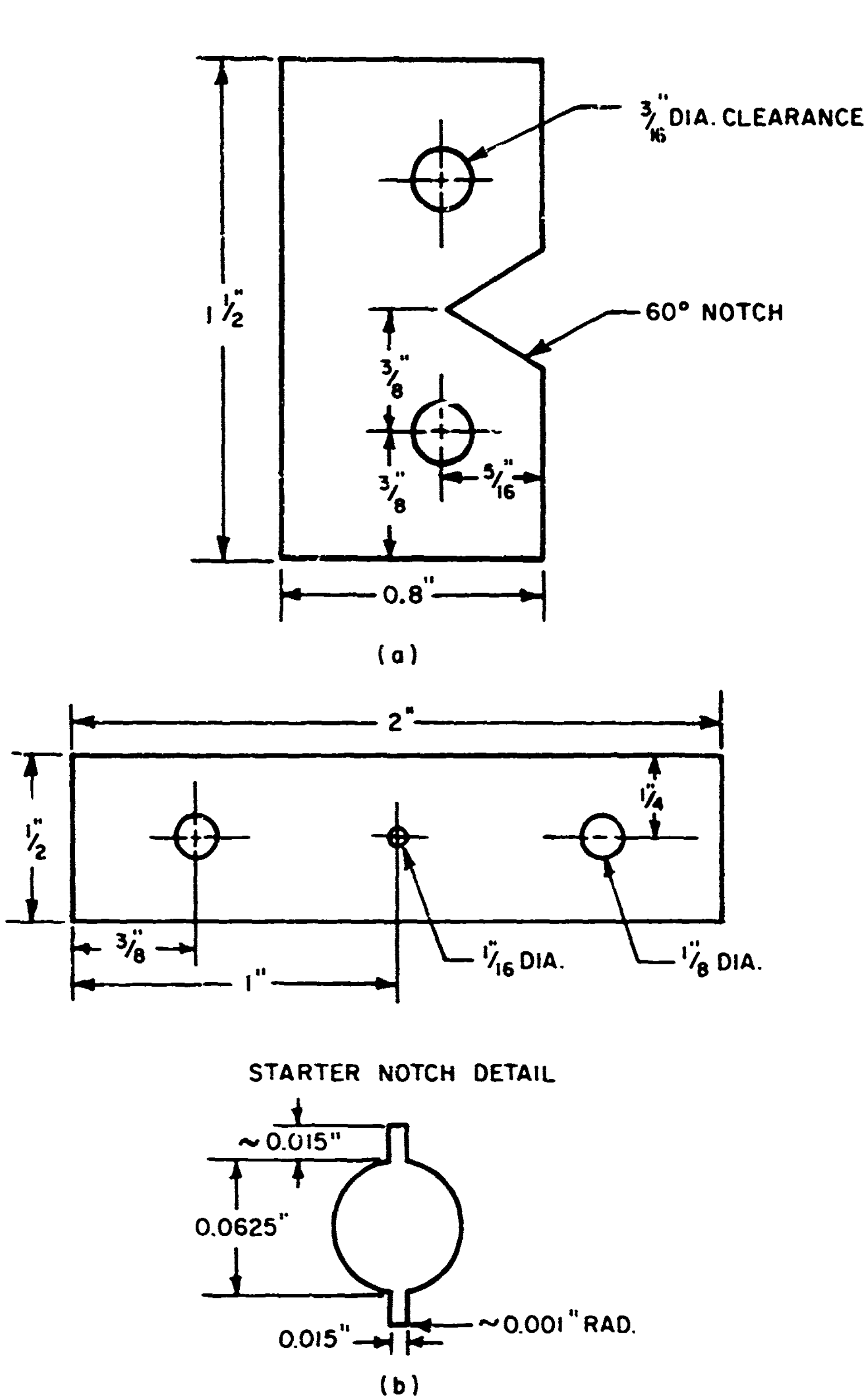


Fig. 3-3. Test Samples Used in Toughness Studies: (a) Tearing Test; (b) Fatigue Crack Propagation Test.

TABLE 3.2

TENSILE DATA FOR NOTCHED GRADE 3 SHEET SAMPLES*

Grain Size (microns)	Condition	k_t	Yield Stress (ksi)	Ultimate Stress (ksi)	NSR	UFE (in.-lb/sq. in.)
5.0	As rec^r	1	62	81	1	-
5.0	"	2.4	64	85	1.05	-
5.0	"	3.0	61	78	0.96	-
5.0	"	3.8	75	94	1.16	-
5.0	"	5.4	170	172	2.12	1100
0.5	As rec^r	1	96	118	1	-
0.5	"	3.0	107	118	1	-
0.5	"	3.8	128	137	1.16	-
0.5	"	5.4	125	129	1.09	1400
0.5	50% PRR	1	125	138	1	-
0.5	"	2.4	130	143	1.04	-
0.5	"	3.0	132	139	1.01	-
0.5	"	3.8	125	133	0.96	-
0.5	"	5.4	193	195	1.41	500

load P the notch root experiences a tensile stress of P/A and a bending stress of 3P/A where A is the cross section at the notch[7]. The notch propagates when the combined stress exceeds the tear strength of the material, i.e.

$$\text{Tear strength} = \frac{P}{A} + \frac{3P}{A} = \frac{4P}{A} \qquad (3)$$

For a notch insensitive material the tear strength is expected to be related to the uniaxial tensile strength while notch sensitive materials should show lower tear strengths. The area under the propagation part of the load-elongation curve defines the tear propagation energy and the unit propagation energy (UPE) is defined as the tear propagation energy

*Tests at room temperature at a nominal strain rate of $3 \times 10^{-4}\ \text{sec}^{-1}$.

divided by the sample cross section. Both the tear strength and the UPE are expected to be dependent on sample size[7]. The sample and procedures adopted here are similar to those employed by Kaufman and Hunsicker [7] for toughness screening and alloy development of aluminum alloys except that the sample size was decreased to allow the use of existing Grade 3 sheet material.

Grade 3 sheet samples with a range of grain sizes were tested at room temperature at a nominal strain rate of 1.1×10^{-2} sec^{-1}. Identical samples of 2024 aluminum alloy in the T3 condition and Ti-6Al-4V in the annealed and STA conditions were tested for comparison. The data obtained are tabulated in Table 3.3. Figure 3-4 shows the variation of tear strength and UPE with yield strength.

For most of the samples tested the root radius of the machined notch was reduced to $\sim$ 0.0005 in. by drawing a razor blade across the notch root. Grade 3 titanium samples in both the recrystallized (5 micron and 0.5 micron) and 50% PRR condition were also tested with as-machined notches ($\sim$ 0.005 in. radius). The results obtained were identical to those observed for samples with the smaller notch radius. The two strongest Grade 3 samples and the Ti-6Al-4V STA material all showed rapid tear propagation. For these samples the UPE was estimated as being less than the elastic energy stored in the test rig which we obtained from the area of the loading curve.

3.2.3 Fatigue Crack Propagation Tests

This test is used to explore the resistance of a material to crack propagation under cyclic loading and consists of measuring the overall crack length 2a as a function of the number of fatigue cycles N. Values of 2a and N are used to obtain values of the crack propagation rate da/dn at specified values of a, which are used to calculate corresponding values of the stress intensity range ΔK, defined as

$$\Delta K = 1.77\Delta\sigma \left\{1 - 0.1\left(\frac{2a}{w}\right) + \left(\frac{2a}{w}\right)^2\right\} a^{1/2} \qquad (3\text{-}4)$$

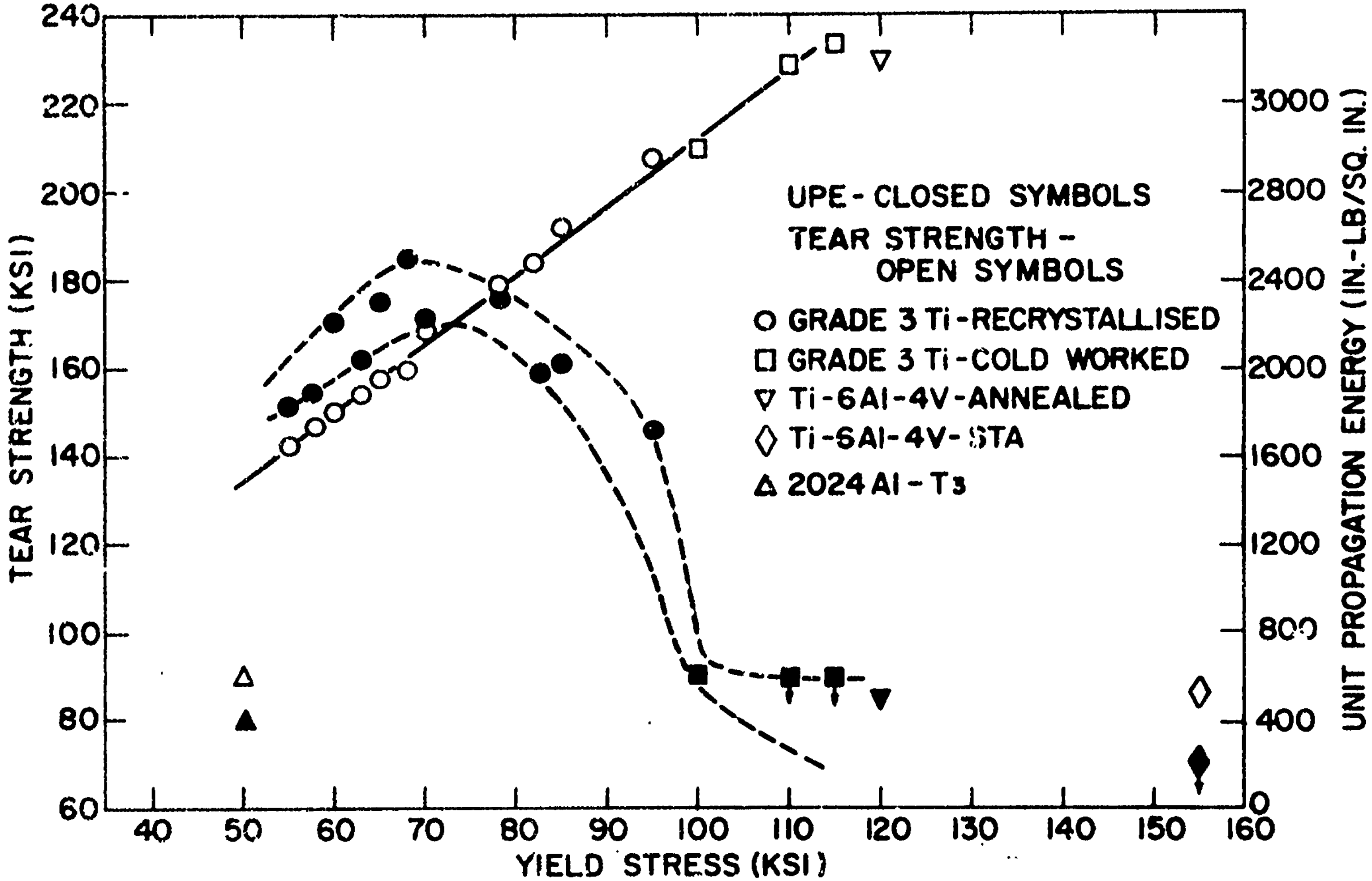


Fig. 3-4. Variation of Tear Strength (Open Points, Solid Line) and UPE (Closed Points, Broken Line) with Yield Strength for 3 Materials in Modified Kahn-Type Tearing Tests.

TABLE 3.3

TEARING TEST DATA FOR GRADE 3 TITANIUM SHEET

Material	Condition	Grain Size (micron)	Yield Strength (ksi)	Tear Strength (ksi)	Unit Propagation Energy (in.-lb/sq in.)
Grade 3 Ti	Rec[r]	20	55	142	1820
"	"	10	58	146	1890
"	"	7	60	150	2200
"	"	4	63	153	2030
"	"	3.5	65	157	2300
"	"	2.8	68	159	2450
"	"	2.2	70	168	2210
"	"	1.2	78	179	2340
"	"	1.9	82	184	1980
"	"	0.8	85	192	2020
"	"	0.5	95	208	1720
	Recovered	-	100	210	610
"	"	-	110	229	<600
"	PRR	-	115	234	<600
2024 Al	T3	-	50	90	400
Ti-6Al-4V	Annealed	-	120	230	510
Ti-6Al-4V	STA	-	155	86	<200

where w is the sample width and $\Delta\sigma$ is the nominal stress range. This form of the stress intensity is believed[8] to be more accurate that the older tangent relation[9-11] for the case of the finite width center-cracked plate sample. The effect of material variables can then be assessed by comparing values of da/dN at the same value of stress intensity (or by comparing the stress intensity required to generate the same growth rate). Although this is an experimentally tedious test it is attractive in that the parameter measured does appear to have some fundamental significance. For example Krafft[12,13] has had quite notable success in predicting crack propagation rates in terms of the basic tensile properties of materials i.e. the strain rate sensitivity and strain hardening rate.

Testing to date has been confined to longitudinal samples of Grade 3 material in three conditions, 7 micron as-recrystallized, 0.5

micron as-recrystallized and 50% PRR as-worked. The sample used, which is shown in Figure 3-3b, is a 1/2 inch wide, 1/16 inch thick sheet sample with a 3/4 inch gage length loaded with pin and wedge grips. The load cycle is a saw-tooth, tension-zero, and the cycle rate is ∿ 10/minute. For a tension-zero fatgue cycle the strain intensity range ΔK is equal to the stress intensity at the maximum applied stress σ_{max}. In the center of the gage is a 1/16 diameter hole. To ensure rapid crack initiation a 0.01 in. wide jeweler's saw is used to extend the circular hole 10-15 mils in both directions perpendicular to the tensile axis. Based on the minimum radius of curvature of the saw cuts, the keyhole notch represents a stress concentrator of a factor of ∿15.

Crack size measurements are started at a crack length of 0.1 inches and continued up to the calculated crack size at which the stress on the remaining section exceeds the yield stress. Fracture is found to follow this point within 100 additional cycles. The crack size is measured under load, using a traveling microscope, to an accuracy of about ± .0005 in. Best resolution of the crack was found to be provided by using very oblique, diffuse (neon) illumination. A double cantilever displacement gage, similar in type to those commonly used in fracture toughness testing has been designed and constructed. This gage clips into the central 1/16 in. diameter hole and would be used to determine the crack size by calibrating the crack size against the displacement produced by a constant applied load. The problem is to design a measurement procedure which will allow a single calibration curve to be used for samples of different yield strengths undergoing varied loading cycles. Although such a procedure has now been designed in concept, extensive experimental checks will be required before it can be applied with confidence, so for the present the direct optical measurement of crack size is preferred. Since crack size is measured at the surface it is important to have some knowledge of the shape of the crack front. This can be obtained from either a scanning electron fractography study of the shape of the fatigue striations or by heat tinting partially cracked samples, followed by fracture and examination. Both techniques will likely be used in the future.

Tests have been performed for each of the three material conditions at several stresses in the range 30-60 ksi so as to extend the obtainable stress intensity range and permit comparison of behavior at constant stress intensity. A typical growth curve is shown in Figure 3-5. Most of the data will be reduced digitally, using a curve-fit program to compute values of da/dN at specified values of a. The program which will also compute values of ΔK is written but not yet debugged. Preliminary data obtained graphically are tabulated in Table 3.4. The samples were all tested at a nominal maximum applied stress of 40 ksi and the fatigue lifetimes were 2102, 3554 and 4546 cycles for the 7 micron, 0.5 micron and PRR samples respectively.

TABLE 3.4

VARIATION OF FATIGUE CRACK GROWTH RATE WITH STRESS INTENSITY AND MATERIAL CONDITION FOR GRADE 3 TITANIUM

Condition	Grain Size	Yield Strength	Crack Growth Rate (micro inches/cycle) at Stres Intensities of			
			16.1	17.9	19.6	21.4
	(microns)	(ksi)	(ksi $\sqrt{in.}$)	(ksi $\sqrt{in.}$)	(ksi $\sqrt{in.}$)	(ksi $\sqrt{in.}$)
As Rec^r	7	60	17	22	31	44
AS Rec^r	0.5	95	12	18	23	27
PRR	-	115	11	15	20	25

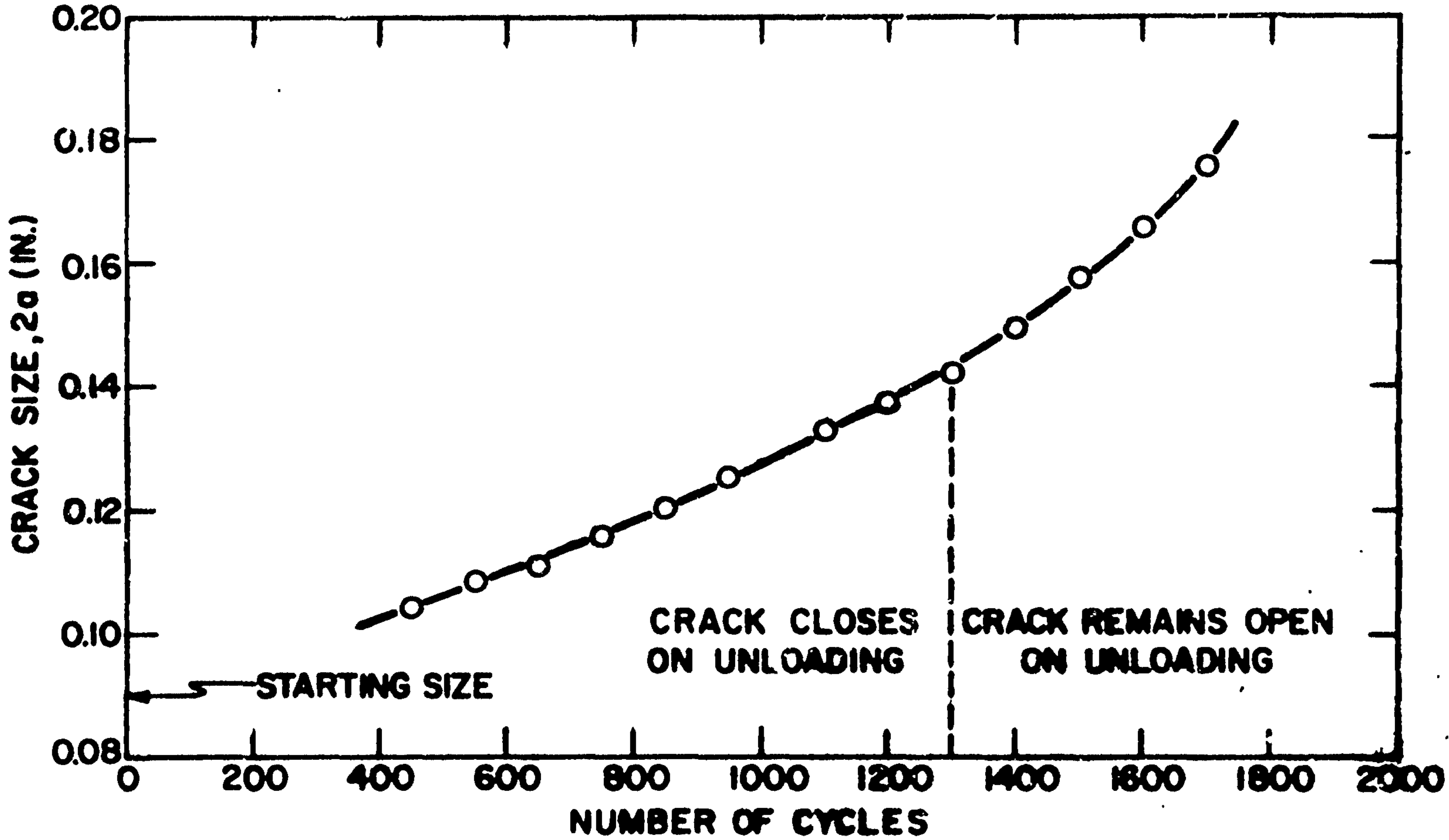


Fig. 3-5. Typical Cyclic Crack Growth Behavior for a Grade 3 Titanium Center-Cracked Sheet Sample.

4. DISCUSSION AND CONCLUSIONS - TITANIUM ALLOYS

4.1 Grain Growth Kinetics

The grain growth data shown in Figs. 2.2 and 2.4 suggest an isothermal grain growth relation of the type

$$d^2 - d_o^2 = (at)_T \tag{4-1}$$

where d is the grain size and t is the annealing time in seconds. Values of the slopes, a, have been computed from Fig. 2.4 which indicates small values of the starting grain size d_o in all cases.

Assuming that the temperature dependence of the grain growth rate is exponential in form we can express the grain growth kinetics by a simple relation of the form

$$d^2 - d_o^2 = At\ e^{-Q/RT} \tag{4-2}$$

where Q is the activation energy of the process and T is the absolute temperature. Hence, combining 4-1 and 4-2 and taking natural logarithms we have

$$\log_e a = -\frac{Q}{RT} + \log_e A \tag{4-3}$$

where A, the pre-exponential factor, is a measure of the new grain boundary area created per unit time.

The present data for $\log_{10} a$ are plotted against 1/T in Fig. 4-1. Also plotted are comparable relations for Grade 4 and iodide purity

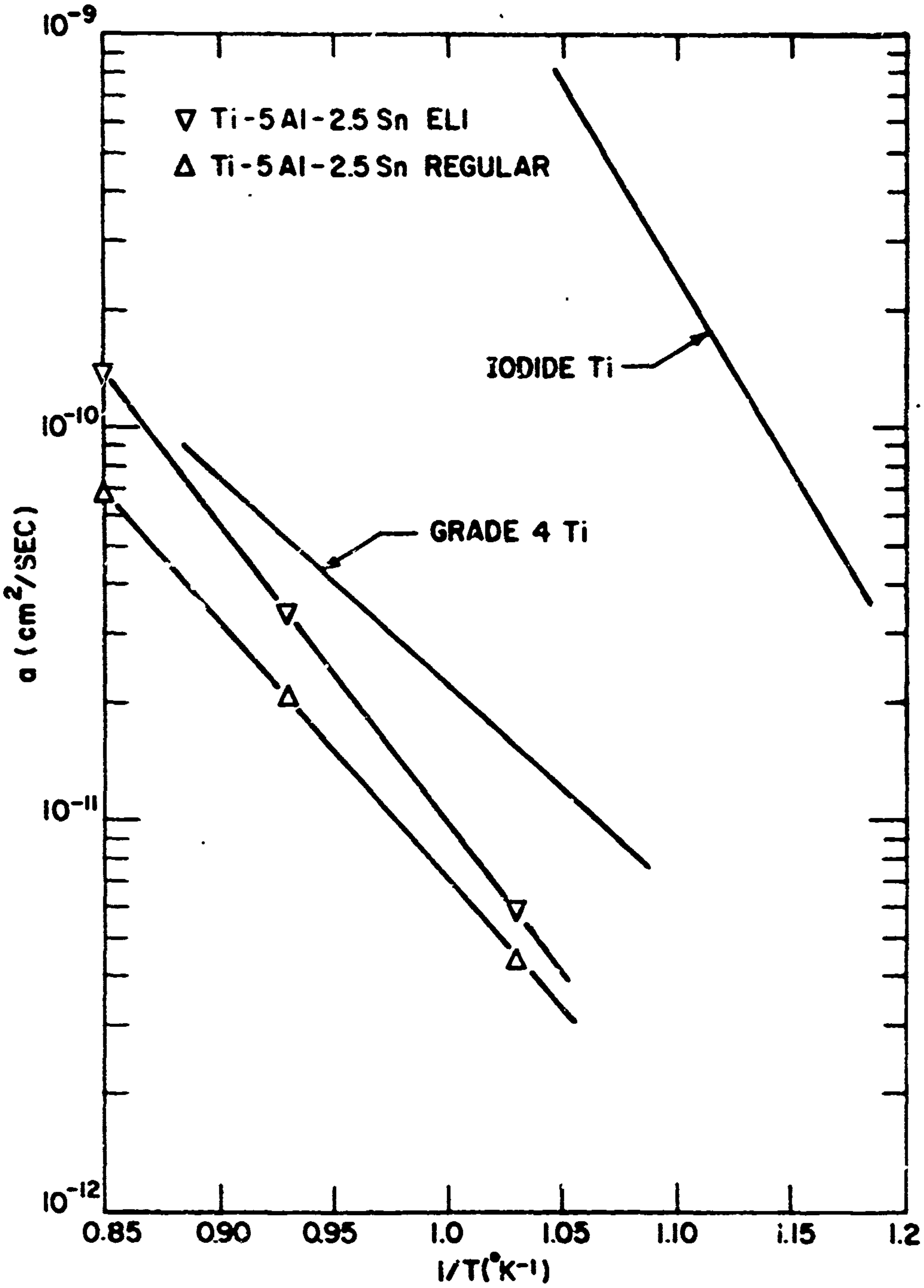


Fig. 4-1. Comparison of the Temperature Dependence of the Grain Growth Rate of Ti-5Al-2.5Sn of Two Purities with the Behavior Observed in Previous Work (4,14) for Unalloyed Titanium.

Grade 4 and iodide purity unalloyed titanium from the data of Jones *et. al.*[4] and Spangler and Herman[14] respectively. The present data are in good agreement with a linear relation and thus confirm equation 4-2. Values of Q and A obtained from the slope and intercept of Fig. 4.1 respectively are tabulated in Table 4.1 together with comparable values for unalloyed titanium of four purities deduced previously[15].

Table 4.1

VALUES OF THE ACTIVATION PARAMETERS FOR GRAIN COARSENING FOR TI-5Al-2.5SN AND UNALLOYED TITANIUM

Material	Total Interstitial Content (ppm)	Q Kcal/gm atom	A Cm^2/sec
Zone refined	120	40	100
Iodide	160	39.5	27
Grade 2 (special)	500	23	1.5×10^{-3}
Grade 4	3000	23	2.4×10^{-6}
Ti-5Al-2.5Sn (Reg)	2500	31	3.2×10^{-5}
Ti-5Al-2.5Sn (ELI)	1270	34	3.6×10^{-4}

From the data tabulated it is evident that the effect of impurities and alloying additions on the coarsening process is quite complex. The unalloyed titanium data show that Q and A both decrease with increasing interstitial content. However, the effect of A must predominate since the experimental observation is that the coarsening rate decreases with increasing interstitial content. The data for the alloys, which show slower coarsening kinetics than any of the unalloyed materials, indicate that substitutional alloying mainly affects the activation energy since the values of A are close to those expected based on the interstitial contents alone. Thus the slow kinetics in the alloys result from the superposed effects of the interstitials (low A) and the substitutionals (high Q).

4.2 Room Temperature Tensile Behavior

4.2.1 Effect of Grain Size

Fig. 2.5 indicates that the effect of grain size on the yield strength of Ti-5Al-2Sn follows the commonly observed Hall-Petch[16,17] relation, vis:

$$\sigma_y = \sigma_i + kd^{-1/2} \tag{4-4}$$

Values for σ_i, the friction stress and k, the Hall-Petch constant, for materials of the two purities are tabulated in Table 4.2 together with comparative values for unalloyed titanium sheet and wire deduced in previous work[1,2,15].

Table 4.2

HALL-PETCH PARAMETERS FOR UNALLOYED TITANIUM AND TI-5Al-2.SN ALLOYS

Material	Total Interstitial Content (ppm)	σ_i (ksi)	k (ksi-mm$^{1/2}$)
Grade 2	1100	36	1.1
Grade 3	1450	47	1.2
Grade 4	3800	65	1.4
Ti-5Aℓ-2.5 Sn (Reg)	2500	99	1.35
Ti-5Aℓ-2.5 Sn (ELI)	1270	[illegible]	1.2

The data in Table 4.2 show that 5% Al and 2.5%Sn in solid solution in titanium increase the friction stress σ_i but have little effect on the Hall-Petch slope k which depends on interstitial content in a similar manner to that previously observed in unalloyed material. In a detailed investigation of the origin of the grain size dependence of the strength of unalloyed titanium[16] it was concluded that the friction stress σ_i could be identified with the short range component of the yield stress which is believed to result from the interaction between dislocations and individual interstitial atoms[4,15,17,18]. The long range component, which in unalloyed material contains the effects of strain and grain size, was shown[16] to be due principally to dislocation interactions, the grain size effect arising because of an interdependence of dislocation density and grain size. In order to assess the effect of substitutional solid solution additions on this model we have performed change-of-strain rate tests so as to obtain values for the activation volume of the short range interaction. Values obtained were 15-20b^3 and 25-30b^3 for regular and ELI purity materials respectively and were essentially independent of strain and grain size. These values are similar to those observed in unalloyed materials with the same interstitial contents and support the belief that the presence of the substitutional alloying elements increases the long range component of the yield stress without effecting the short range component or the thermally activated deformation mechanism. A similar conclusion was reached by Sargent and Conrad[19] in stress relaxation experiments on Ti-4Al. Thus in the α-alloys the friction stress σ_i contains a short range component due to the dislocation interactions and a long-range component of 35-40 ksi due to the presence of the substitutional solutes.

The data in Table 2.5 indicate that grain size refinement into the 0.5 micron range increases the yield strength of Ti-5Al-2.5Sn by 40-50 ksi depending on interstitial content. The strain hardening rate decreases at fine grain sizes with the consequence that the uniform elongation is reduced by about a factor of 2 and the increase in the ultimate strength (20-30 ksi) accompanying grain refinement is therefore less than the yield stress increase. As observed for unalloyed material the R.A. increases slightly with grain refinement but in contrast to unalloyed behavior the total elongation at failure is somewhat decreased by grain refinement. Ductility remain quite adequate, however, and the decrease in elongation appears to be more than offset by the increase in yield strength attained.

4.2.2 Effect of PRR

Reference to the data in Table 2.5 indicates that 40% PRR raises the yield strength by about 30 ksi beyond the strength level attained in the recrystallized samples. Curiously, the increase is larger for the ELI purity material which generally exhibits lower strain hardening rates. The increase in the UTS is similar but is accompanied by a substantial decrease in uniform elongation ($\sim$ a factor of 3) and total elongation (up to a factor of 2). Although the strength levels attained are impressive in themselves it is felt that the rather low elongations may not prove adequate in this case, particularly in view of the effects of PRR on the toughness of unalloyed material discussed elsewhere in this report.

4.3 Thermal Stability and Age Hardening Behavior

The short term, static thermal stability limit appears to be about 600°C for both materials in the 40% PRR condition, which is the recrystallization temperature. This is a similar result to that obtained previously for unalloyed material[2]. It is expected, on the basis of the behavior of unalloyed material, that the dynamic thermal stability limit will prove to be somewhat lower than this.

The hardness increase observed for annealing temperatures near 500°C is worthy of further comment. Such a hardness increase could be due to a strain aging effect, or to interstitial pickup during annealing (thermal stability anneals were performed in air) or to strengthening due to the precipitation of a second phase. Strain aging is not considered to be likely explanation since the present temperature range is well above that in which strain aging behavior is observed in unalloyed material. A similar hardness increase was observed in the recrystallization studies where anneals were performed in vacuum (see Fig. 2.2) so that any explanation based on interstitial pickup appears untenable. Furthermore such an explanation would have difficulty explaning a hardness peak since the rate of contamination would be expected to continuously increase with increase in temperature. The precipitation explanation seems to fit the observations well. Observation of a hardness peak with aging time as well as with aging temperature is characteristic of an age hardening system[20] and a 5% Al content may exceed the solid solubility of Al in Ti below 600°C,[21] although the Ti-Al binary phase diagram is still somewhat in dispute[22]. A sample which was recrystallized at 700°C then annealed at 500°C did not show any significant change in hardness, indicating that the presence of a high dislocation density is necessary to promote the hardening mechanism, possibly suggestive of heterogeneous nucleation of precipitates on dislocations. Another possible explanation lies in the recent observation of short range order hardening in Ti-Al binary alloys[23].

Whatever the underlying mechanism, the important point is that a significant hardening does occur on annealing cold deformed Ti-5Al-2.5Sn at 500°C and that the maximum hardnesses observed are equivalent to yield strengths of ≥200ksi for both ELI and regular purity material. From the point of view of the overall objectives of the program it is essential to find out whether or not the hardness increase is reflected in a usable improvement in tensile properties i.e. is the material embrittled? Tensile samples are currently being fabricated to investigate this question.

4.4 Summary of Conclusions for the Ti-5Al-2.5Sn Alloy Study

The major conclusions of this section of the program may be summarized as follows:

1. Grain coarsening kinetics for Ti-5Al-2.5Sn alloys are slower than those of unalloyed titanium with comparable interstitial contents. The substitutional alloying additions principally influence the activation energy for grain growth. The pre-exponential factor in the grain growth equation depends upon interstitial content in alloys in a similar manner to that observed in unalloyed titanium so that ELI purity material shows more rapid grain growth than regular purity material.

2. Grain size refinement leads to an increase in yield strength according to the Hall-Petch equation. Strength increases of 40-60 ksi have been obtained accompanied by some decrease in uniform elongation. The substitutional alloying additions have

no effect on the Hall-Petch slope which depends on interstitial content in a similar manner to that observed previously in unalloyed titanium. The friction stress is increased by 30-40 ksi compared with unalloyed material due to the presence of the substitional additions. This increase is athermal(long range) in nature and the presence of the solutes appears to have no effect on the rate controlling thermally activated deformation mechanism at room temperature.

3. The yield strength of the fine grain size samples may be increased further by post recrystallization reduction (PRR). 40% PRR results in a strength increase of $\sim$ 30 ksi accompanied by a further substantial decrease in elongation. The strongest samples tested showed yield strength of $\sim$ 180 ksi (ELI) and > 190 ksi (Regular) with reduction in area of > 50% in both cases.

4. The short term static thermal stability limit of the 40% PRR microstructure is close to the recrystallization temperature, as observed for unalloyed material.

5. Annealing of deformed alloy wires at about 500°C produces a hardness increase which has the general characteristics of age hardening. The nature of the hardening mechanism has not yet been established and it remains to be seen whether or not the observed hardness increase corresponds to a useful strength enchancement.

5. DISCUSSION AND CONCLUSIONS - UNALLOYED TITANIUM SHEET

5.1 The Notched Tensile Test

The results of the notched tensile tests presented in Table 3.2 were completely discussed in a previous report[2] where the observation of notch strength ratios (NSR) > 1 for the range of stress concentration investigated was interpreted in terms of the combined effects of strain rate and plastic constraint. It was concluded that increase of strength by grain refinement and/or PRR was accompanied by some increase in notch sensitivity. As discussed in Section 3.2.1, since the NSR does not explicitly contain the absolute stress level it is not clear how to relate this parameter to toughness for materials with widely different strengths. Table 3.2 also contains values of a new parameter, the unit fracture energy (UFE) for samples in three thermomechanical conditions containing the most severe notches investigated. It is believed that this parameter may be a more significant toughness indicator for materials with differing yield strengths. It is probably unwise to generalize from so few data but the results obtained to date suggest that grain size refinement increases the UFE slightly while PRR results in a substantial decrease. This is a similar trend to that observed in the tearing tests discussed in the next section. It is planned to extend this study in further work so as to investigate the effects of more severe stress concentrators on materials covering the entire grain size and substructure spectrum.

5.2 The Tear Test

The tear test data obtained during the present program are presented in Table 3.3 and Fig. 3.4. The Figure indicates that the tear strength is directly related to the yield strength for the unalloyed titanium samples in all thermomechanical conditions, which is the expected result for a notch insensitive material. Note that the data point for the Ti-6Al-4V alloy in the annealed condition lies close to the unalloyed line while the data points for the aluminum alloy and the Ti-6Al-4V in the STA condition lie far below it, indicative of their substantially greater notch sensitivity. The data for the UPE, which are also plotted on the graph, are quite interesting. Data for unalloyed titanium in the recrystallized condition show a broad maximum in the yield stress range 70-80 ksi. Unalloyed titanium samples in the PRR conditions show markedly lower values than those of recrystallized samples, as do the aluminum alloy and the titanium alloy in both heat treatments. The value for the Ti-6Al-4V alloy in the annealed condition is close to that for the unalloyed titanium sample in the PRR condition with no recovery treatment, which has a similar yield strength.

Attempts have been made in the past to correlate the UPE with the plane strain fracture toughness K_{Ic} for aluminum alloy samples(7). Although the fundamental basis for any such correlation is somewhat

with increasing UPE as shown by the data in Table 5.1.

Table 5.1

CORRELATION OF UPE WITH K_{IC} FOR THE EXPERIMENTAL MATERIALS

Material	Condition	UPE (in.-lb/sq in.)	K_{Ic} (Typical) (ksi $\sqrt{in.}$)
Grade 3 Ti	Recrystallized (10-20μ)	1850	>80
2024 Al	T3	400	40
Ti-6Al-4V	Annealed	510	50
Ti-6Al-4V	STA	<200	30

It thus appears that the value of K_{Ic} for recrystallized samples of all grain sizes is probably > 80 ksi $\sqrt{in}$ while 50% PRR reduces this to ~ 50 ksi $\sqrt{in}$.

To further illustrate the effect of grain requirement, the yield stress and UPE are plotted against the inverse square root of the grain size in Fig. 5-1. The error bars on the UPE data are based on the experimentally observed scatter in duplicate tests. Scatter in the yield strength data was typically ± 1 ksi. While the yield stress data show the familiar Hall-Petch relation with grain size (σ_i = 47 ksi, k = 1.1 ksi-mm$^{1/2}$) the UPE data again show a broad maximum, centered in the range 20-30 mm$^{-1/2}$ corresponding to a grain size of 1-2 microns. We have attempted to illustrate the practical consequences of this result in Fig. 5-2. In this graph we have considered a hypothetical application in which it is desired to employ Grade 3 sheet with an optimum combination of strength and toughness, these two requirements being equally weighted. To determine the optimum grain size we have plotted grain size in Fig. 5.2 against combined strength plus toughness parameter, calculated as the sum of the relative yield stress (yield stress/maximum observed yield stress) and the relative UPE (UPE/maximum observed UPE). This parameter rises initially with decreasing grain size and reaches a plateau at a value of about 1.72 for grain sizes below 1 micron, indicating that materials with grain sizes below this are equivalent in the hypothetical application. If strength was given a higher weighting than toughness the finest grain size material attainable would be optimum, while for a higher toughness weighting the optimum grain size would lie in the range 1-2 microsn.

While a detailed explanation of the variation of the UPE with grain size and cold work has not been attempted, some insight can be gained by considering that the UPE may be regarded as a parameter which combines strength and ductility. The pertinent uniaxial paramters are probably the yield strength (or UTS) and the uniform elongation and a combination of these parameters appears capable of explaining the variation of the UPE. We have seen previously that grain size refinement initially increases the yield strength with little change in uniform elongation. Below about 3 microns the uniform elongation begins to decrease significantly as the yield stress increases and PRR results in a rapid decrease of uniform elongation with a comparatively small increase of strength. As a first attempt at a quantitative correlation we have simply multiplied the yield stress by the uniform elongation in selected cases to

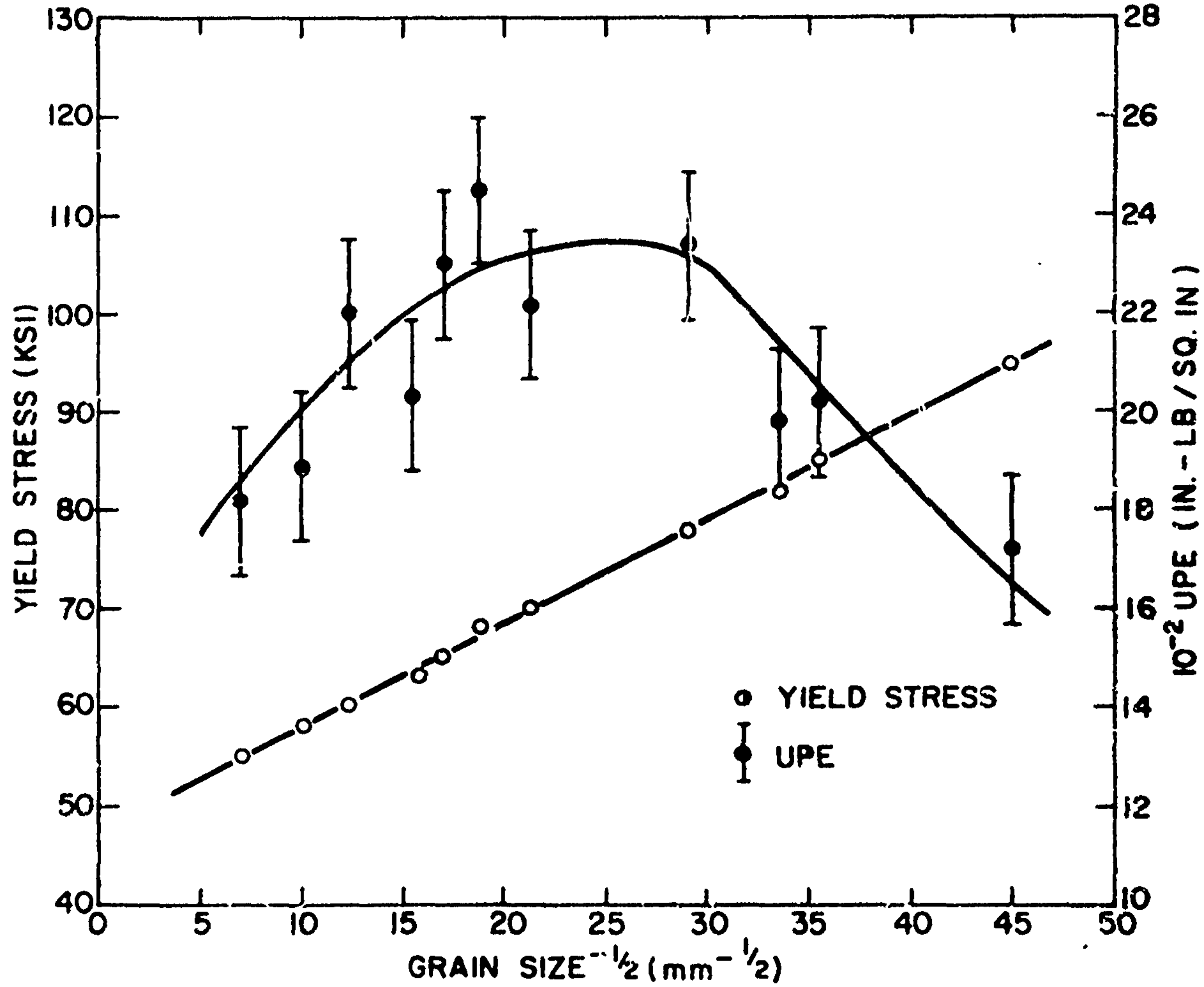


Fig. 5-1. Showing the Effect of Grain Size on the Yield Stress and Unit Propagation Energy for Grade 3 Unalloyed Titanium.

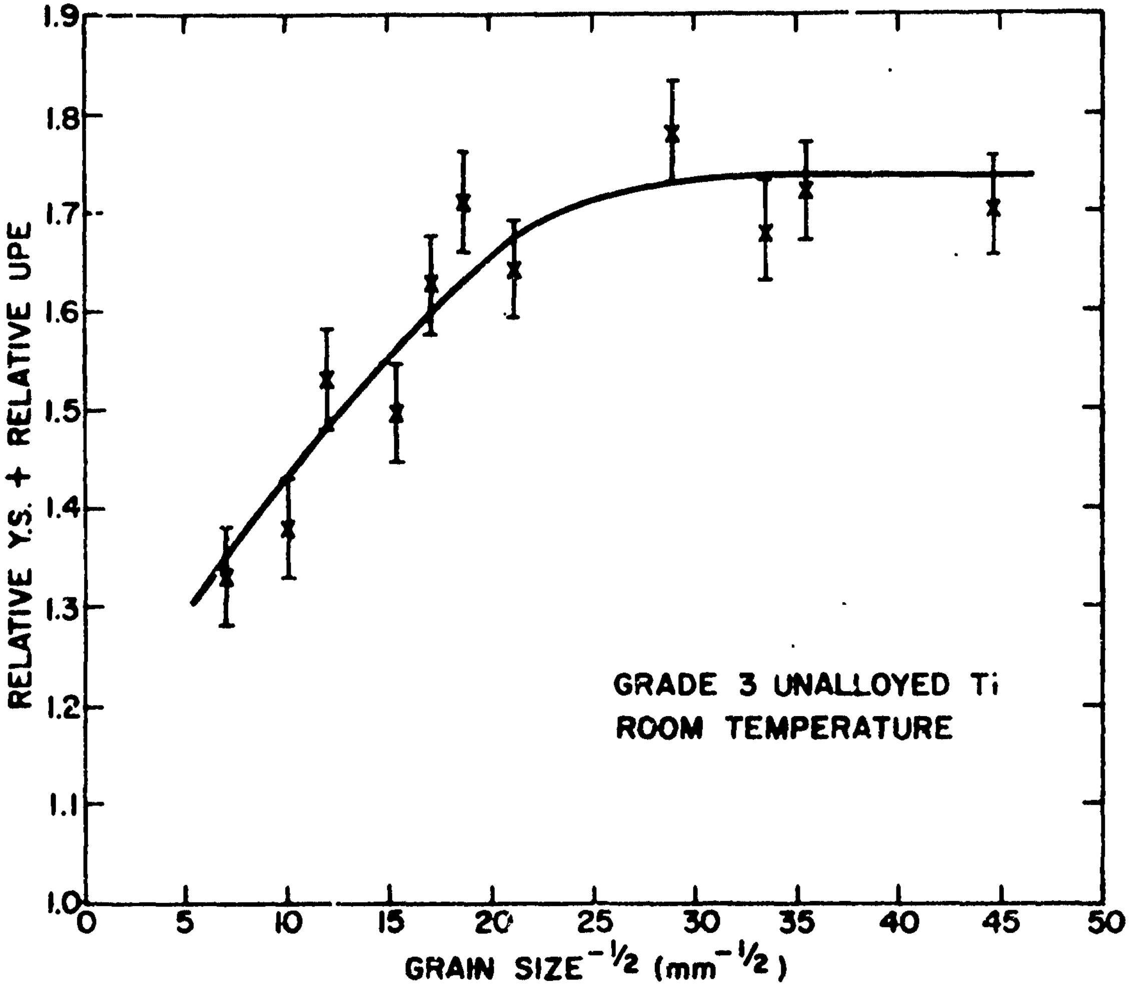


Fig. 5-2. The Variation of a Parameter Combining Yield Strength and Tear Toughness with Grain Size for Grade 3 Unalloyed Titanium at Room Temperature.

obtain a pseudo energy per unit volume parameter. The data are shown in Table 5.2 and it is seen that the uniaxial tensile parameter varies in a suprisingly similar manner to the UPE for Grade 3. Notice however that the same rrelation does not apply to the Ti-6Al-4V alloy which shows much smaller values of UPE than those expected on the basis of the tensile parameter. Notch sensitivity is probably the necessary factor omitted from this simple model so that the tensile and tear data only correlate well if the material is essentially insensitive to the stress concentration due to the tear notch. The simple relation between tear strength and yield stress (Fig. 3.4) indicates that this is probably the case for the Grade 3 samples but not for the Ti-6Al-4V samples.

Table 5.2

CORRELATION OF UNIAXIAL TENSILE DATA AND THE UPE

Material	Condition	σ_y (ksi)	e_u (in./in.)	$\sigma_y \times e_u$ (in.-lb/cu in.)	UPE (in.-lb/sq in.)
Grade 3	20 microns	55	0.18	9900	1820
Grade 3	1.2 microns	78	0.16	12500	2340
Grade 3	0.5 microns	95	0.1	9500	1720
Grade 3	50% PRR	115	0.025	2900	<600
Ti-6Al-4V	Mill Anneal	120	0.08	9600	510
Ti-6Al-4V	STA	155	0.06	9300	<200

5.3 Fatigue Crack Propagation Tests

The fatigue crack growth rate data shown in Table 3.4 indicate that the growth rate is a sensitive function of stress intensity and is dependent also on material condition. Krafft[12] predicts a fourth power relation between growth rate and stress intensity range (which for tension-zero fatigue is the stress intensity at the maximum applied stress). The present data are presented on a log plot in Fig. 5-3. The range of stress intensity covered by the data available at present is too small

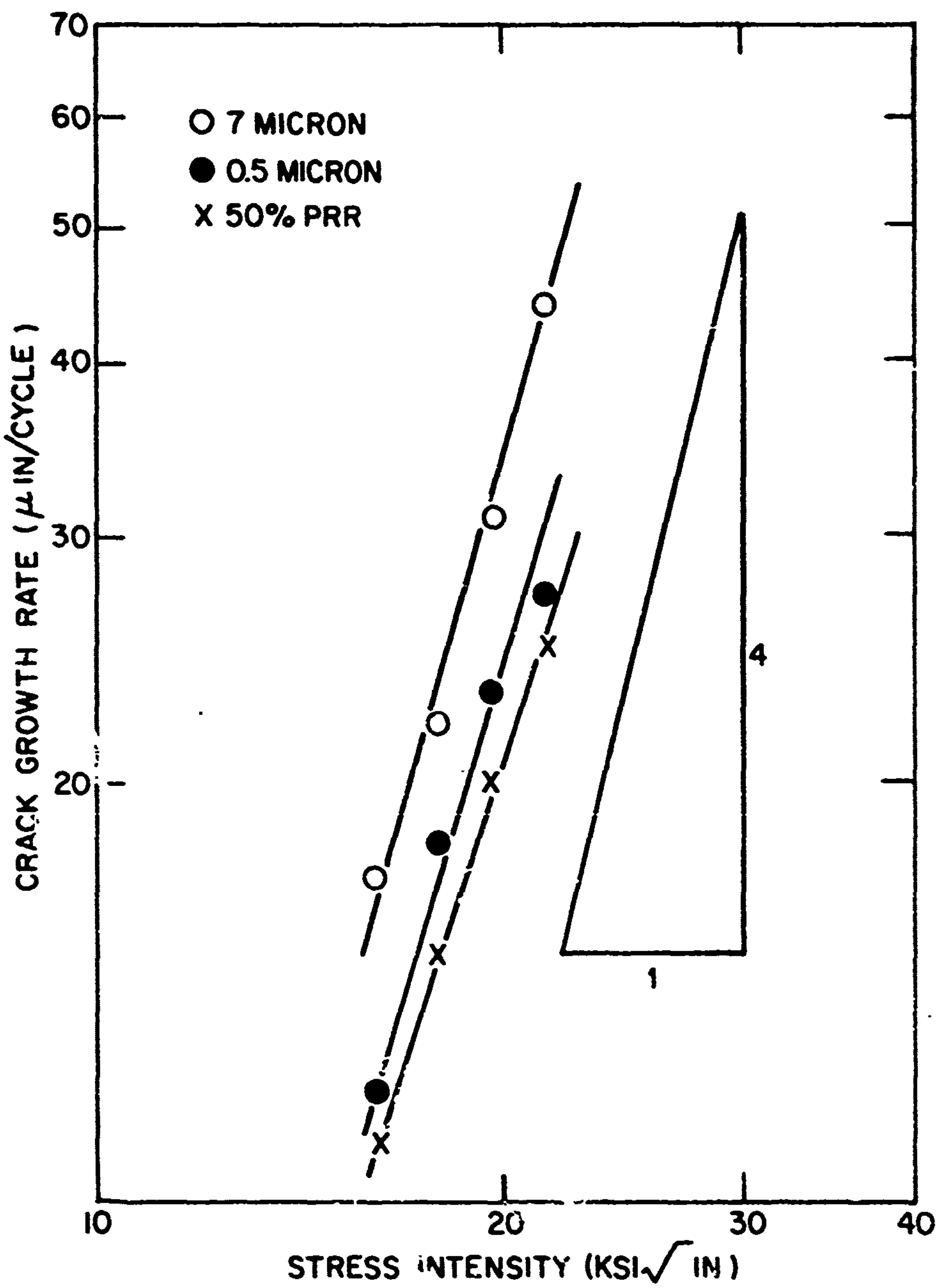


Fig. 5-3. Variation of Fatigue Crack Growth Rate with Stress Intensity Range for Grade 3 Unalloyed Titanium in Three Conditions at Room Temperature.

for any definite conclusion but Fig. 5-3 certainly supports a dependence of crack growth rate on a high power of ΔK and is not inconsistent with the fourth power relation proposed by Krafft[12].

The crack growth rate at a constant stress intensity decreases as the strength increases, whether the strength increase is due to grain refinement or to cold work. Again, there are too few data for any conclusion to be drawn with certainty but the data are not inconsistent with a simple inverse relation between crack growth rate and yield stress. The fatigue lifetimes are more sensitive to yield stress than are the crack growth rates. This follows simply from the fact that stable crack growth to a greater size can occur in the higher strength materials provided that they are not significantly more notch sensitive. Rough calculations were made of the stress on the remaining gage section when fracture occurred. For all three material conditions this stress was found to be close to the tensile UTS, indicating essentially notch insensitive behavior.

5.4 Summary of Conclusions for the Unalloyed Titanium Sheet Program

The major conclusions for this section of the program may be summarized as follows:

1. Grain refinement and cold work reduce the notch resistance of Grade 3 titanium to some extent as indicated by the notch strength ratio. However, under all conditions investigated the material continues to exhibit notch insensitive properties. On the basis of a very limited number of data, grain refinement appears to improve the unit fracture energy of Grade 3 titanium in notched tensile tests while cold work reduces this parameter.

2. In modified Kahn-type tearing tests a direct relation between yield strength and tearing strength is observed for Grade 3 titanium irrespective of the thermomechanical condition. Other materials investigated show inferior tearing strengths at a given yield stress than those predicted by the Grade 3 titanium irrespective of the thermomechanical condition. Other materials investigated show inferior tearing strengths at a given yield stress than those predicted by the Grade 3 titanium relation. The unit propagation energy for tear failure shows a complex relation to yield stress. Recrystallized samples show very

large values of this parameter in comparison to the other materials investigated and a maximum is observed in the 1-2 micron grain size range. Cold worked samples show much smaller values than recrystallized samples. It is possible to account qualitatively for the observed behavior in terms of a combination of the uniaxial tensile yield stress and uniform elongation for Grade 3 titanium but not for Ti-6Al-4V alloy.

3. The fatigue crack propagation rate measured for a constant stress intensity range decreases systematically with increasing yield stress for Grade 3 titanium irrespective of the thermomechanical condition. The crack propagation rate is a sensitive function of the stress intensity range as observed in other materials. Fatigue lifetimes are dependent on yield strength to a greater extent than is predictable from the decreased crack propagation rate alone, and final fracture occurs when the remaining section stress approaches the uniaxial UTS, indicating notch insensitive behavior.

6. AREAS FOR FURTHER STUDY

6.1 Titanium Alloy Study

This program has demonstrated that grain size refinement and cold work effect the room temperature tensile properties of Ti-5Al-2.5Sn wires in much the same manner as that observed previously for unalloyed titanium. The fatigue and toughness behavior of the thermomechanically strengthened alloy should be investigated to assess the changes produced in these service-relevant properties and the tensile properties above and below room temperature should be documented. The possible use of the apparent age hardening phenomenon observed for annealing temperatures near 500°C should be investigated and its origin elucidated. Possible methods for transferring the optimum thermomechanical processing parameters to the fabrication of a sheet product should be explored.

Only preparatory work on β-alloys was carried out during the present program. Further work is required with the objective of at least establishing the effect of grain size on the tensile behavior of one or more β-alloys.

6.2 Unalloyed Titanium Sheet Study

The continuing study is aimed at investigating in depth the effects of material variables, (grain size, impurity content, thermomechanical condition, texture, etc.) on the toughness of unalloyed titanium sheet under various types of loading. Notched tensile, tear, and fatigue crack propagation tests should be continued and the scope of the investigation widened to include variables not considered to date. The results obtained during the present program have been extremely encouraging in that they indicate that strengthening unalloyed titanium by grain size refinement and/or cold work does not result in catastrophic reduction of any of the toughness-related parameters investigated, and in some cases leads to significant improvements (e.g. tear strength, fatigue crack propagation rate).

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