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Metallurgical Characterization of Thermomechanically Processed U-0.75 wt.% Ti

Chester V. Zabielski

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13. ABSTRACT (Maximum 200 words)

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The objective of this study was to develop higher strength U–0.75 wt.% Ti by thermomechanical procedures. The approach was to replace the conventional solution treated, quenched, and aged (STA) process for U–0.75 wt.% Ti with warm rolling and warm or cold swaging. The effect of working on structure, hardness, tensile properties, compressive strength, and fracture toughness was determined. Deformation strengthening of previously hot-extruded and slow-cooled U–0.75 wt.% Ti was found to significantly increase the hardness, the tensile and compressive yield strengths, the ultimate tensile strength, and the reduction in area. There was no appreciable change in fracture toughness. The combinations of strength, ductility, and toughness obtained by deformation strengthening of this as-extruded material were generally inferior to those characteristic of STA processing. U–0.75 wt.% Ti, which was solution treated, water quenched, and warm rolled to a large reduction and then cold swaged, achieved the highest values in hardness, tensile and compressive yield strength, and ultimate tensile strength. Fracture toughness values were comparable to the conventionally processed alloy and reduction in area values were significantly greater. Deformation strengthening of solution treated and quenched material resulted in substantially better combinations of strength, ductility, and toughness than those characteristic of STA processing.

1. Introduction

The work reported herein describes a program designed to determine the influence of metallurgical and thermomechanical process variables on the mechanical behavior of the U–0.75 wt.% Ti alloy in the following conditions: as hot-extruded; hot-extruded and warm worked; and solution treated, water quenched and warm worked. Specific objectives of this investigation were to develop thermomechanical treatments to impart high strength and ductility.

In prior work [1] U–0.75 wt.% Ti bars were extruded from 88.9 to 20.3 mm in diameter as a function of extrusion temperature between 537 and 871°C in 56°C increments and fully characterized. It was found that the finest grain size and the overall best as-extruded mechanical properties were produced at 732°C. Follow-on extrusion work [2] between 662 and 746°C in 14°C increments confirmed that 732°C was indeed the overall optimum extrusion temperature. Tensile ductility was lower than in the prior study [1], probably due to hydrogen pickup during the molten salt heat treatment which was required for best temperature control. Further, the (0.2%) yield strength obtained did not meet the minimum requirement; namely, 724 MPa. By comparison, conventional treatment requires solution treating at 850°C and aging at 355°C to achieve the desired strength level and elongation.

In order to retain, or further refine, the small grain size achievable by the 732°C extrusion, and to increase the yield strength and elongation, a follow-on rod rolling operation was employed. Justification for this approach is based upon a study [3] which showed that isotropic mechanical properties are retained by unalloyed uranium which was rolled unidirectionally and that warm deformation, up to 80% reduction, produced an increase in both fracture stress and tensile ductility of unalloyed uranium.

Eckelmeyer [4,5] found that cold working the water quenched martensitic phase in U–0.75 wt.% Ti produces a significantly higher elongation and reduction in area when compared with identical yield strength materials produced by aging treatments. Since the as-quenched U–0.75 wt.% Ti martensitic phase provided the highest ductility available for this alloy, Eckelmeyer rolled as water quenched 15.2 mm thick plate up to 40% reduction. Rolling was carried out at temperatures up to 380°C. A determination was made of the

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effects of deformation and aging on hardness. Identical rates of hardening due to deformation were observed at all temperatures up to 300°C. When deformation was carried out at higher temperatures combined deformation and age hardening effects occur, thus shifting curves to higher hardness. Post-deformation aging also results in additional hardening. The hardening effects of deformation and aging were found to be additive.

Significant residual stresses are introduced by the initial water quenching. However, small amounts of post quenching room temperature deformation are currently used to decrease the magnitudes of these residual stresses. Small amounts of deformation make the radial residual stress gradient smaller and reduces macrosurface differences. At higher strains, localized deformation was found to occur at approximately 20% reduction regardless of deformation temperature and its extent increased with increasing reduction at all temperatures [4]. This is potentially undesirable because small regions of highly deformed material could act as initiation sites for stress corrosion cracks.

2. Procedure

2.1. Materials and processing

U–0.75 wt.% Ti material had the chemical analysis: 0.72 wt.% Ti, 37 ppm C, 50 ppm Fe, 10 ppm Al, 10 ppm Cu, 55 ppm Si, 10 ppm Ni, < 10 ppm Nb, remainder uranium consistent with a good quality heat. This material was in the form of 20.3 mm in diameter bar which was obtained by double extruding a 305 mm diameter ingot at 871°C to billets 86.4 mm in diameter and subsequently extruding at 732°C to size. The heat-up for the 732°C extrusion was accomplished in either of two ways: brought to temperature in molten salt; glass coated and heated in air to reduce hydrogen pickup. The molten salt heat treated billets were heated to 850°C in vacuum in the gamma phase region to remove hydrogen to less than 0.10 ppm prior to quenching and warm rolling or cold swaging operations. Table 1 lists processing summaries (conditions 1A through 6A) in column 2 and total overall reduction by warm working from 20.3 mm diameter for conditions 2A through 5A is listed in column 3.

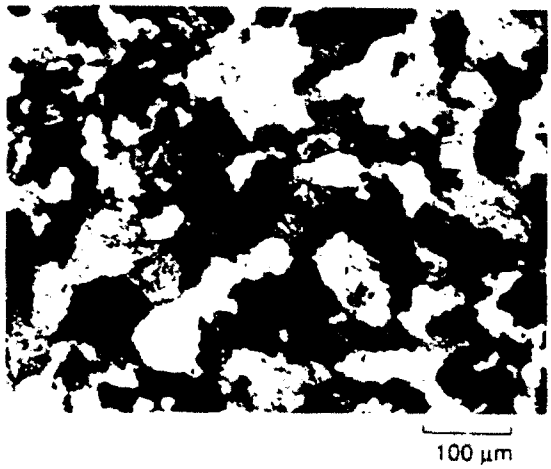
More specifically, conditions 1A, 2A, and 6A were extruded from 86.4 mm diameter billets to 20.3, 20.3 and 14.5 mm diameter bar, respectively, from a molten salt bath. For condition 2A, the as-extruded 20.3 mm in diameter bar was further solution treated at 850°C in vacuum for 2 h, water quenched and warm rolled 63.1% at 300°C, and then cold swaged 19.5% to 11.1 mm in diameter for a total reduction of 70.4% of the original cross-sectional area.

Conditions 3A, 4A, and 5A were extruded using 86.4 mm diameter glass-coated billets to 20.3 mm. For condition 3A, the as-extruded bars were warm rolled 75.7% to 10.0 mm in diameter. For condition 4A, the

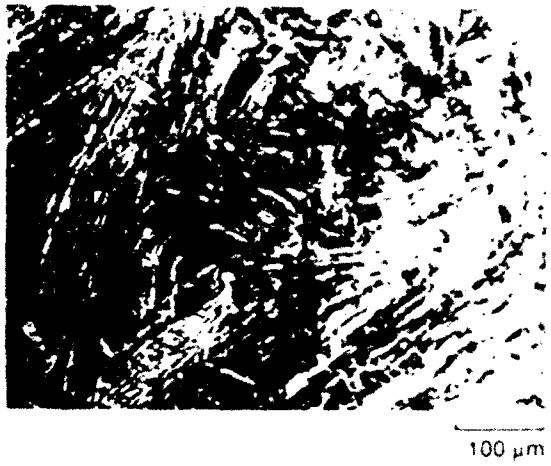
Table 1
Comparison of mechanical properties [a] of as-extruded and warm worked bars with differing processing histories. As received: extruded 732°C – conditions 1A through 5A to 20.3 mm diameter; condition 6A to 14.5 mm diameter. Processing notes: ST – solution treated 850°C; WQ – water quenched; WR – warm rod rolled 300°C; CS – cold swaged – 1.27 mm; DWS – double warm swaged 300°C

Cond.	Proc.	Tensile (0.2%)							Compressive 0.2% YS (MPa)	K_Q		Hardness	
		RDN (%)	Total RDN (%)	YS (MPa)	UTS (MPa)	Elon. (%)	RA (%)	Mod. of Elast (GPa)		21°C (MPa $\sqrt{m}$)	−46°C (MPa $\sqrt{m}$)	HRC Trans.	HRC Longit.
1A	As received	0	0	504	1028	8.3	9.3	167	613	41	34	29.3	28.4
2A	ST	0	70.4	1598	2058	9.8	37.4	154	1076	69	40	44.6	46.5
	WQ + WR + CS	63.1 19.5											
3A	WR	75.7	75.7	889	1625	5.9	17.1	144	845	30	21	39.2	42.0
4A	WR + DWS	48.9 21.4 + 18.7	67.4	995	1498	6.0	14.9	154	827	48	37	39.4	39.4
5A	WR + WS	58.9 18.6	66.5	1259	1967	4.6	13.8	151	898	42	33	38.7	42.5
6A	As received	0	0	587	1142	7.9	9.2	148	697	40	32	31.4	31.4

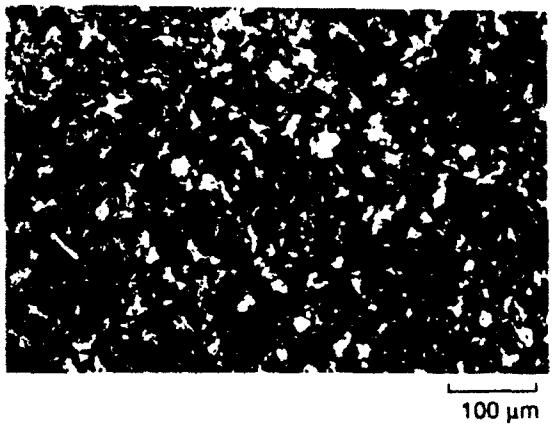
[a] Each value – average of minimum of two test specimens.



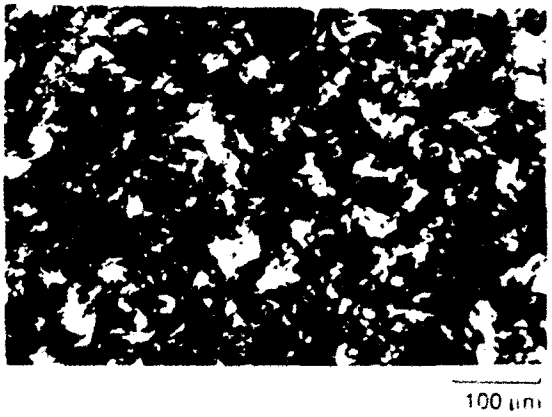
1A Extruded to 20.3 mm diameter bar Equiaxed α phase



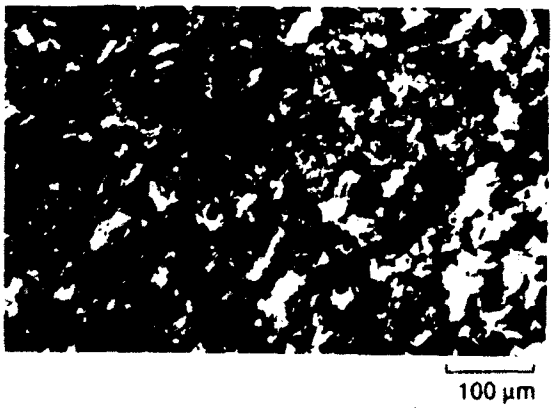
2A Solution treated at 850°C for two hours in vacuum, water quenched, warm rolled at 300°C and cold swaged 70.4% to 11.1 mm in diameter. α' phase martensite



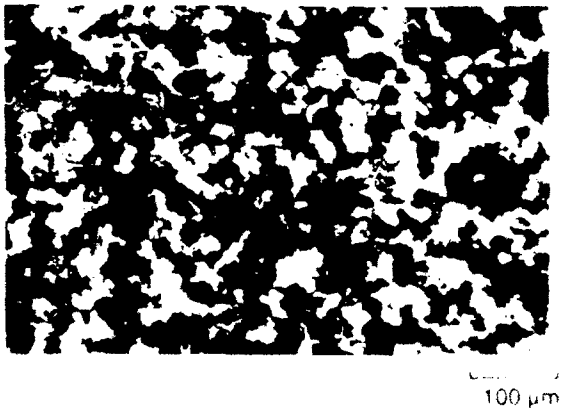
3A Warm rolled 75.7% at 300°C to a diameter of 10.0 mm. Fine structure α phase.



4A Warm rolled and double warm swaged 67.4% at 300°C to a diameter of 11.6 mm. Fine structure α phase.

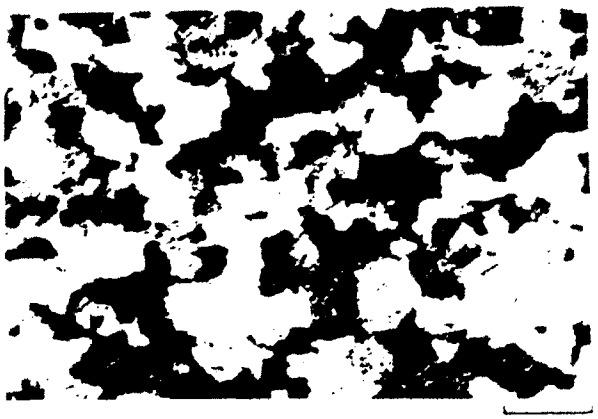


5A Warm rolled and single warm swaged 66.5% at 300°C to a diameter of 11.8 mm. Fine structure α phase.



6A Extruded to 14.5 mm diameter bar Equiaxed α phase

Fig. 1. Section transverse to extrusion direction at the center of the bar. Extrusion temperature 732°C. Unetched polarized light.



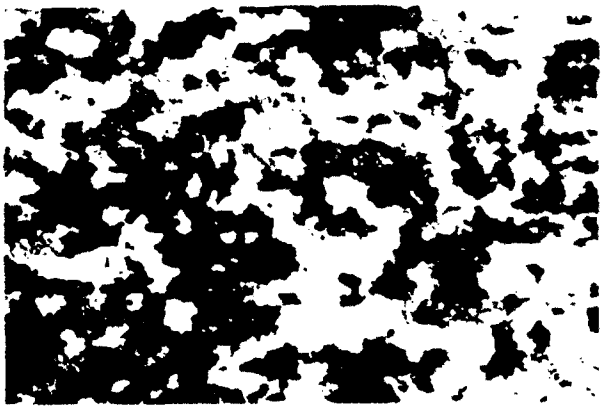
1A Extruded to 20.3 mm diameter bar. Equiaxed α phase.

2A Solution treated at 850 C for two hours in vacuum, water quenched, warm rolled at 300°C and cold swaged 70.4% to 11.1 mm in diameter. Elongated α' phase martensite.

3A Warm rolled 75.7% at 300°C to a diameter of 10.0 mm. Elongated fine structure α phase.

4A Warm rolled and double warm swaged 67.4% at 300°C to a diameter of 11.6 mm. Elongated fine structure α phase.

5A Warm rolled and single warm swaged 66.5% at 300 C to a diameter of 11.8 mm. Elongated fine structure α phase.

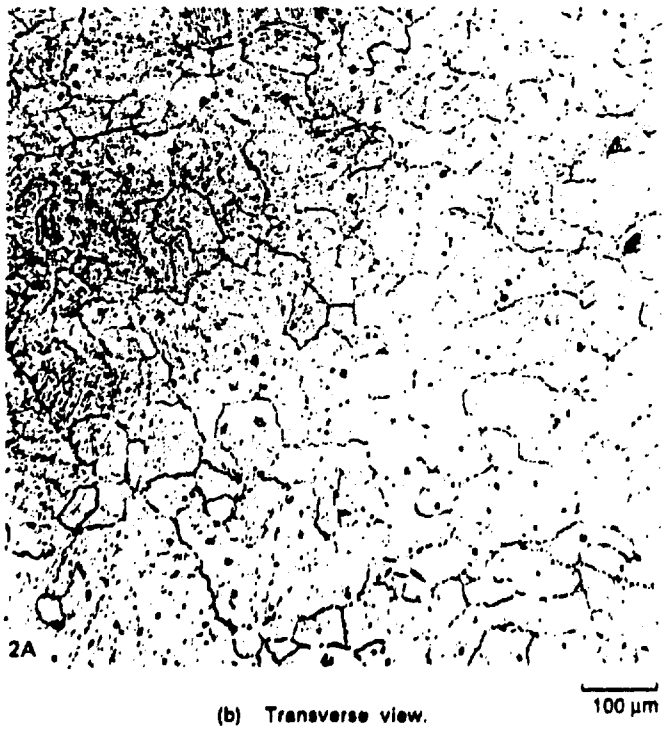


6A Extruded to 14.5 mm diameter bar. Equiaxed α phase.

Fig. 2. Section parallel to extrusion direction at the center of the bar. Extrusion temperature 732 C. Unetched polarized light.

as-extruded bars were warm rolled and double warm swaged 67.4% to a diameter of 11.6 mm. For condition 5A, the as-extruded bars were warm rolled and single warm swaged 66.5% to 11.8 mm diameter bar. All warm rolling and warm swaging was conducted at 300°C.

(a) Longitudinal view.



(b) Transverse view.

Fig. 3. Solution treated 850°C for two hours, water quenched, warm rolled at 300°C and cold swaged 70.4% to 11.1 mm in diameter. α′ phase martensite. Etched bright field.

2.2. Sampling – specimen preparation

Slow-bend V-notch Charpy impact specimens (type A) [6] were utilized for fracture toughness measurements. Type A Charpy impact specimens were machined from larger diameter bars > 12.7 mm in diameter. The dimensions were 10.0 mm × 10.0 mm × 55.0 mm. For the smaller diameter warm worked bar < 12.7 mm in diameter, the machined dimensions were 7.50 mm × 7.50 mm × 55.0 mm. Both types of specimens were used for static fracture toughness measurements. The notches were always located on surfaces lying nearer the outer diameter of the bar. Prior study [7,8] disclosed a similarity between the K_{IC} and K_Q values obtained, thus, it was decided to use only the simplest and least costly specimen, the V-notch bend Charpy impact specimen, and report K_Q values for the materials.

The tensile specimens from the larger diameter bars > 12.7 mm were 73.0 mm long with gauge diameter of 6.40 mm and gauge length of 25.4 mm; for the smaller diameter warm worked bar < 12.7 mm, the 57.2 mm long specimens had gauge diameter of 4.06 mm and a gauge length of 16.3 mm. The compression specimens had an $L/D = 2$.

2.3. Fracture toughness test method

The procedure [9] for making K_Q measurements involved three-point bend testing of notched Charpy specimens that had been precracked in fatigue. Load versus displacement data was recorded autographically. The maximum load in each test was recorded and the nominal crack strength was determined from this value using the original dimensions of the specimen with the single beam bending equation. The Rockwell C hardness of each specimen was measured by taking the average of four equally spaced readings on the back of each specimen.

3. Results and discussion

3.1. Microstructure

The unetched microstructures for the extruded and warm worked bars are shown under polarized light for the six conditions 1A through 6A in figs. 1 and 2 for

Table 2
Mechanical properties of U-0.75 wt.% Ti bars – identically solution treated at 850°C 2 h; vertically water quenched at 0.46 m per minute; aged 16 h at 355°C in gas recirculating furnace

No.	0.2% YS (MPa)	UTS (MPa)	Mod. of E (GPa)	Elon. (%)	RA (%)	Hardness (HRC)	
						L [a]	T [b]
01	806	1380	149	17	23	42.1	39.7
79	817	1380	162	20	28	41.1	38.7

[a] L – longitudinal section.
[b] T – transverse section.

the transverse and longitudinal sections, respectively. Conditions 1A and 6A extruded at 732°C to 20.3 mm and 14.5 mm diameter showed an equiaxed α phase grain structure with an ASTM grain size of six and eight, respectively. Condition 6A had a significantly smaller grain size than condition 1A. Conditions 3A, 4A, and 5A extruded at 732°C and subsequently warm worked had an α phase structure with indistinct grain boundaries. The longitudinal section exhibited markedly elongated grains.

Condition 2A, which was solution treated at 850°C, water quenched, warm rolled and cold swaged, shows for the transverse view under polarized light an α′ phase martensitic structure. The corresponding longitudinal view shows a highly elongated martensitic structure. The etched structure of condition 2A in fig. 3 under bright field examination discloses a duplex grain structure with areas of coarse and fine prior-gamma grains for the transverse view. The predominant area of fine prior-gamma grains has an ASTM grain size of 5. The longitudinal view showed highly elongated prior-gamma grains with indistinct grain boundaries.

3.2. Tensile tests

A summary of tensile test results is shown in table 1. It was found that the ultimate tensile strength and (0.2%) yield strength for condition 2A, extruded at 732°C, solution treated, quenched, and warm rolled and cold swaged were outstanding and significantly exceeded those for the other five conditions. Conditions 3A, 4A, and 5A which were extruded at 732°C, warm rolled or warm swaged were lower. Conditions

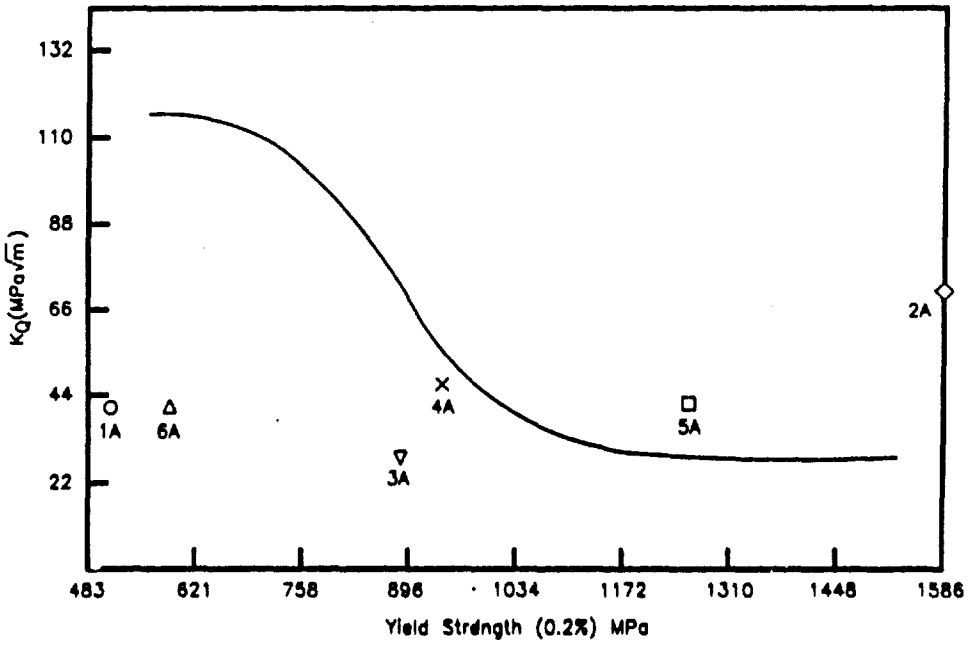


Fig. 4. Fracture toughness of bars extruded at 732°C and warm worked at 300°C superimposed on a ratio analysis diagram for U-0.75 wt.% Ti.

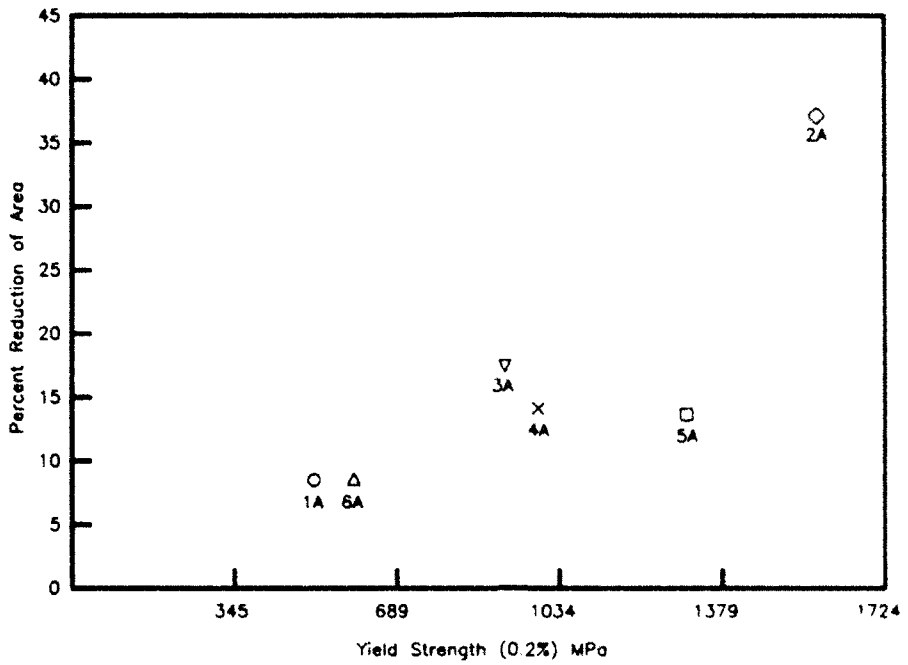


Fig. 5. Reduction in area values versus yield strength (0.2%) for bars extruded at 732°C and warm worked at 300°C

1A and 6A which were extruded at 732°C and not subsequently warm rolled or swaged had the lowest values. Since condition 2A also had a significant elongation, almost 10%, at a very high strength level and fracture toughness values which were comparable to currently produced water quenched and aged U–0.75

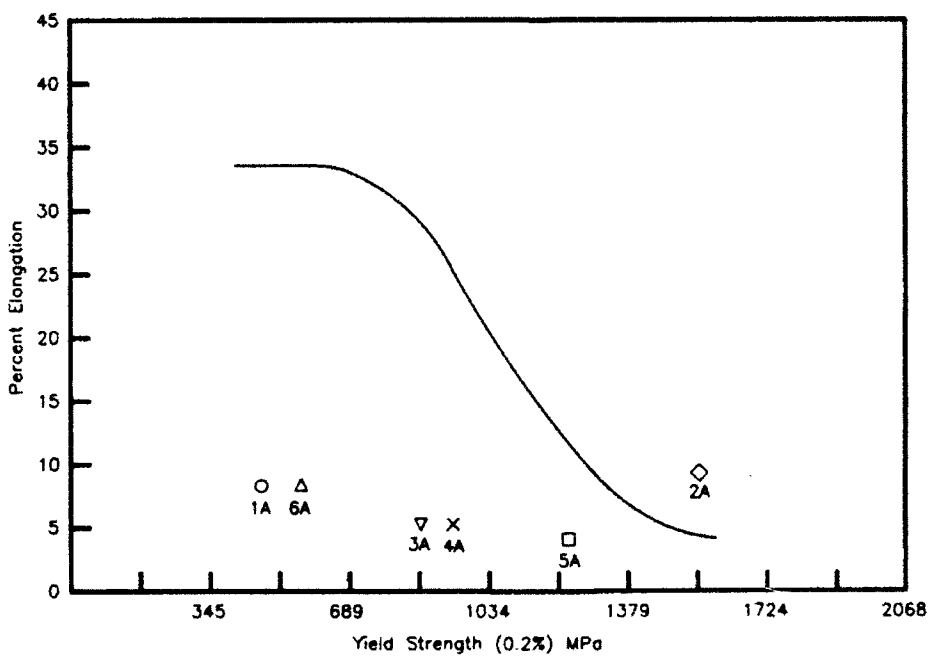


Fig. 6. Elongation values for bars extruded at 732°C and warm worked at 300°C superimposed on ratio analysis diagram for U–0.75 wt.% Ti.

wt.% Ti alloys, a detailed mechanical properties evaluation was carried out and is presented in the latter part of this paper. For comparison purposes, mechanical properties were also determined on bars from the same heat that had been identically commercially heat treated; i.e., solution treatments were given with aging. These properties are shown in table 2.

3.3. Fracture toughness versus yield strength (0.2%)

Fig. 4 plots room temperature fracture toughness versus yield strength (0.2%) superimposed on a ratio analysis diagram [8] containing a technological limit line which represents the highest values of fracture resistance previously measured for U–0.75 wt.% Ti as-extruded and STA alloys. As-extruded at 732°C conditions 1A and 6A had similar K_Q Charpy fracture toughness values of 41 and 40 MPa $\sqrt{m}$, respectively, which were well below the technological limit. Condition 6A had the higher yield strength of 597 MPa, and smaller grain size. Condition 3A, extruded at 732°C and warm rolled 75.7% at 300°C had a K_Q value of 30 MPa $\sqrt{m}$ also below the technological limit. Conditions 4A and 5A extruded at 732°C, warm rolled and warm swaged at 300°C, had fracture toughness K_Q values of 48 and 42 MPa $\sqrt{m}$, respectively. The latter value exceeded the technological limit at the yield strength of 1259 MPa. If warm rolling was followed by warm swaging, an appreciable reduction in fracture toughness did not occur with an increase in yield strength. Condition 2A, extruded at 732°C, solution treated at 850°C water quenched, warm rolled at 300°, and cold swaged 70.4% had room temperature fracture toughness K_Q value of 69 MPa $\sqrt{m}$ at high yield strength of 1598 MPa, markedly exceeding the technological limit.

3.4. Reduction in area and elongation versus yield strength (0.2%)

Fig. 5 shows a plot of room temperature reduction in area versus yield strength (0.2%). The elongations plotted in fig. 6 are superimposed on a ratio analysis diagram containing a technological limit line which represents the highest values of percent elongation previously measured. Conditions 1A and 6A extruded at 732°C had reductions in area slightly below 10% and elongations of about 8% at yield strengths below 724 MPa. Conditions 3A, 4A, and 5A as extruded at 732°C and, respectively, warm rolled 75.7% and warm rolled and warm swaged 67.4% and 66.5% had a decrease in percent reduction in area from 17.1 to 13.8, and in elongation from 6 to 4.6 with increasing yield strengths over the range 889 to 1259 MPa. Condition 2A extruded at 732°C solution treated at 850°C, water quenched, warm rolled and cold swaged 70.4% had an outstanding percent reduction in area of 37.4 and an elongation of 9.8 which exceeded the technological limit line in fig. 6. The percent reductions in area for the warm rolled and swaged Conditions 2A through 5A greatly exceeded the elongations indicating significantly more necking occurs for these conditions when compared to as-extruded Conditions 1A and 6A.

3.5. Fracture toughness versus hardness

Table 1 shows room temperature fracture toughness K_Q values and HRC for the as-extruded 732°C conditions 1A and 6A and warm worked conditions 2A through 5A. In comparison to Condition 1A the higher hardness, smaller grain size condition 6A with smaller diameter bar did not exhibit a decrease in fracture toughness. Conditions 4A and 5A which were extruded, warm rolled and warm swaged also did not exhibit a decrease in fracture toughness at a significant increase of Rockwell C hardness to 40. Condition 3A, which was processed similarly to conditions 4A and 5A, did not undergo a final warm swaging procedure and the fracture toughness fell slightly below the required K_Q value 33 MPa $\sqrt{m}$ at −46°C to prevent fracture at low temperature. Solution treated, water quenched, and warm rolled condition 2A had the highest fracture toughness value at the HRC of 46.5. An increase in HRC values for the warm rolled and swaged conditions 2A through 5A did not cause a decrease in K_Q values.

3.6. Fracture toughness versus temperature

Table 1 shows fracture toughness K_Q values at −46°C and +21°C. In condition 2A the solution treated, water quenched and warm rolled condition easily exceeded the required K_Q value of 33 MPa $\sqrt{m}$ at −46°C. Conditions 4A and 5A which were extruded warm rolled and warm swaged also met this requirement. Condition 3A extruded and warm rolled had a value significantly below the specification limit at −46°C. Condition 6A extruded to 14.5 mm in diameter had a value slightly below the requirement.

3.7. Compressive yield strength (0.2%)

Fig. 7 plots compressive yield strength (0.2%) versus tensile yield strength (0.2%) for alloys with different

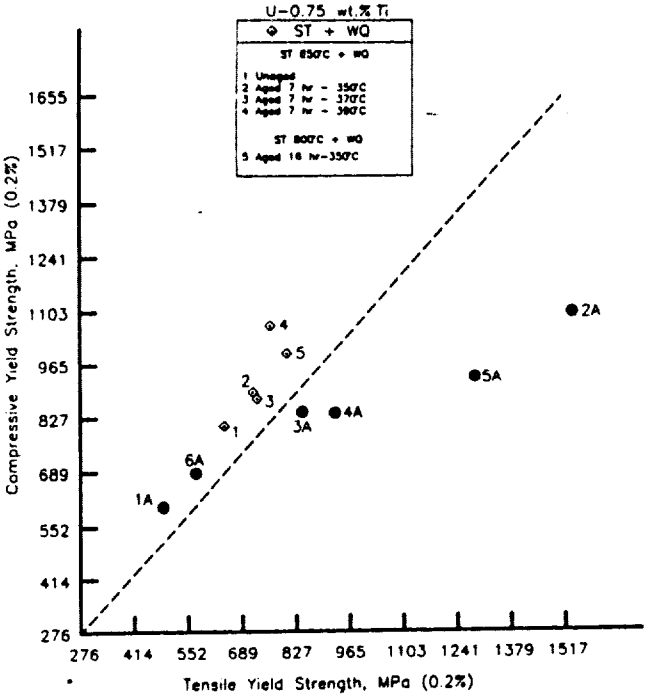


Fig. 7. Compressive yield strength versus tensile yield strength (yield strengths at 0.2% offset).

processing histories. In all cases where the material was not warm or cold worked, the compressive yield strength (0.2%) exceeds the tensile yield strength (0.2%) by 100 to 276 MPa. These materials all have compressive yield strengths which are slightly higher than corresponding tensile yield strengths. This occurs in crystallographically textured uranium and its alloys which deform by combinations of slip and twinning. The preferred orientation favors slip deformation when the alloy is compressed and twinning deformation when the alloy is stressed in tension. Since different stresses are required for slip and twinning, this results in the tensile–compressive yield strength anisotropy. The data for these alloys appear generally above the 45° line of fig. 7.

For conditions 3A, 4A, and 5A, which are extruded and warm worked, the reverse is true: i.e., the tensile yield strengths (0.2%) exceeds the compressive yield strengths (0.2%) and are plotted below the 45° line. For condition 2A which was solution treated, water quenched, warm rolled and cold swaged the tensile yield strength (0.2%) greatly exceeds the compressive yield strength (0.2%). Those alloys for which the tensile yield strength (0.2%) significantly exceeds the compressive yield strength (0.2%) have been found by X-ray analysis to have appreciable preferred orientation. These latter alloys exhibit a pronounced Bauschinger effect which greatly exceeds the textural contribution. Elongation of the alloy in tension beyond

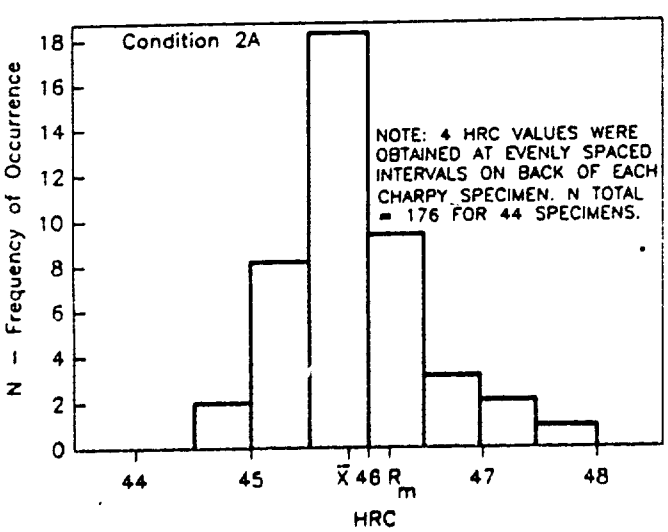


Fig. 8. Histogram – N versus HRC for 20.3 mm diameter U-0.75 wt.% Ti bars, solution treated at 850°C for two hours, water quenched, warm rolled at 300°C, and cold swaged 70.4%.

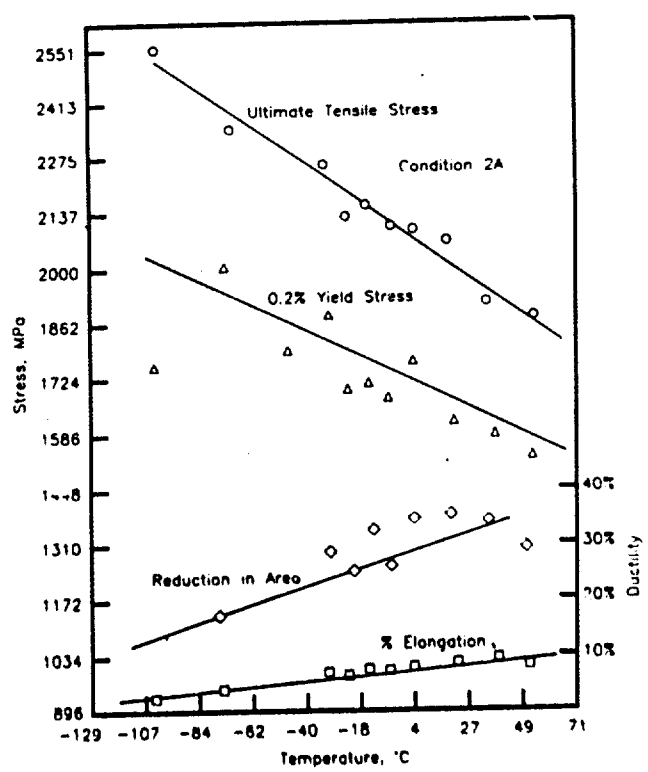


Fig. 9. Variation of tensile properties with temperatures for 20.3 mm diameter U-0.75 wt.% Ti bars, solution treated at 850°C for two hours, water quenched, warm rolled at 300°C and cold swaged 70.4%.

Table 3
Variation of tensile properties with temperature for 20.3 mm diameter U–0.75 wt.% Ti bars – solution treated at 850°C for 2 h, water quenched and warm rolled 70.4% at 300°C, condition 2A

Temperature (°C)	0.2% YS (MPa)	UTS (MPa)	Elon. (%)	RA (%)	Mod. of E (GPa)	HRC
+54	1507	1861	8.1	29.9	174	46.4
+38	1549	1896	9.7	34.5	169	45.7
+21	1598	2058	9.8	37.4	154	45.7
+4	1760	2093	8.4	33.7	160	48.2
−4	1661	2101	7.5	26.6	152	45.3
−2	1702	2148	7.8	31.9	160	45.7
−21	1696	2117	6.9	25.4	161	45.9
−29	1877	2241	7.3	28.3	138	46.2
−46	1784	nd [a]	nd [a]	nd [a]	156	nd [a]
−73	2002	2348	4.5	18.2	159	46.8
−101	1737	2568	3.0	9.3	172	45.5
−129	nd [a]	1129	1.2	1.1	159	nd [a]

[a] Note: nd = no data.

its yield strength and strain hardening by warm or cold working correspondingly decreases its axial compressive yield strength while increasing its tensile yield strength. The tensile and compressive yield strength values for conditions 2A, 3A, 4A, and 5A indicate that the degree of anisotropy increases with rising tensile yield strength level.

3.8. HRC – condition 2A

A histogram, as shown in fig. 8, was obtained by plotting frequency of occurrence versus HRC in intervals of 0.5 units. Values were determined on backs of K_Q Charpy specimens. The small range in HRC disclosed in the histogram indicates the bars are quite uniform in hardness with location with the major part of values falling between 45 to 46.5 HRC. The average HRC value obtained above was 46. The average HRC obtained for a transverse disc specimen was 44.6, disclosing a slight anisotropy in properties.

3.9. Tensile properties versus temperature – condition 2A

Fig. 9 and table 3 illustrate the effect of test temperature on mechanical properties determined over the temperature range −101°C to +54°C. With a decrease in temperature, the ultimate tensile strength increased from 1861 to 2568 MPa and the (0.2%) yield strength increased from 1507 to 2002 MPa. Correspondingly, the percent elongation decreased from 9.7 to 3.0 and the reduction in area decreased from 34.8 to 9.3%. For the same test temperatures the ultimate tensile strength the (0.2%) yield strength and reduction in area exceed the values for the conventionally processed vertically water quenched and aged bars by 50 to 100%. Comparison with room temperature tensile data can be made for conventionally processed bars in table 2.

3.10. Fracture toughness values versus temperature – condition 2A

Fig. 10 and table 4 compares over the temperature range −73 to +38°C the fracture toughness data for

Table 4
Comparison of fracture toughness Charpy K_Q and hardness values for bars 2A and B

Temperature (°C)	2A [a] (MPa $\sqrt{m}$)	B [b] (MPa $\sqrt{m}$)	HRC 2A	HRC B
+38	72	74	45.7	39.4
+21	69	65	45.7	39.5
+4	60	61	46.2	39.7
−12	49	52	45.7	39.7
−21	44	50	46.0	39.6
−29	46	46	46.2	39.4
−46	36	40	–	39.4
−73	38	35	46.8	39.7

[a] 2A: solution treated at 850°C for 2 h, water quenched, warm rolled at 300°C, and cold swaged.
[b] B: solution treated at 800°C for 2 h and 850°C 1/2 h vertically water quenched at 0.46 m per minute, aged 16 h at 350°C in lead bath.

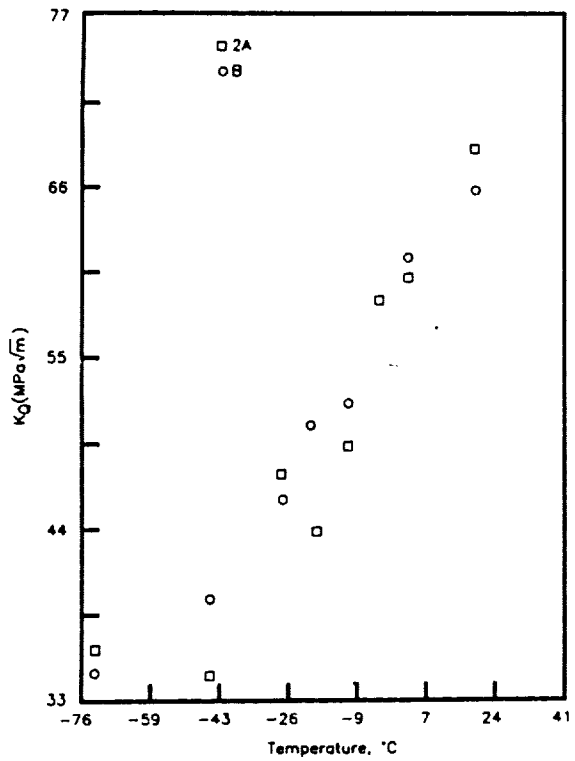


Fig. 10. Comparison of the variation of fracture toughness with temperature for (2A) 20.3 mm diameter U-0.75 wt.% Ti bars, solution treated at 850°C for two hours, water quenched, warm rolled at 300°C, and cold swaged 70.4% and (B) bars solution treated at 800°C for two hours, and 850°C 1/2 hour vertically water quenched at 0.46 m per minute, aged 16 h at 350°C.

the extruded at 732°C solution treated, water quenched, warm rolled cold swaged bar (see condition 2A) with conventionally processed vertically water quenched and aged bars (B). The fracture toughness values were found to be similar although HRC values were higher for condition 2A.

4. Summary

The warm rolling and warm swaging of fine grained as-extruded U-0.75 wt.% Ti at large reductions was found to significantly increase the hardness, reduction in area, the tensile and compressive yield strength, and the ultimate tensile strength. A small reduction occurred in the percent elongation. There was no appreciable change in fracture toughness values.

U-0.75 wt.% Ti which was solution treated, water quenched, warm rolled to large reduction, and cold swaged achieved the highest values in hardness, tensile, and compressive yield strength. When compared to the conventionally processed solution treated, water quenched and aged U-0.75 wt.% Ti, the fracture toughness values were similar, the reduction in area values were significantly greater and the percent elongation was reduced.

This latter processing procedure is recommended over the conventional standard process whenever very high strength U-0.75 wt.% Ti alloys with significant ductility are required.

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