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Effect of silicides on tensile properties and fracture of alloy Ti–6Al–5Zr–0.5Mo–0.25Si from 300 to 823 K

C. RAMACHANDRA, VAKIL SINGH
Department of Metallurgical Engineering, Institute of Technology, Banaras Hindu University, Varanasi 221 005, India

The tensile properties and fracture behaviour of alloy Ti–6Al–5Zr–0.5Mo–0.25Si (wt %) have been investigated over a wide range of temperature from 300 to 823 K, in the as-water-quenched (WQ) and different aged (473 to 1073 K for 24 h) conditions following β-solution-treatment (1323 K for 0.5 h). There is only a limited increase in strength but a drastic reduction in the ductility, at 300 K, due to ageing at $\geqslant$ 923 K. There is strong dynamic strain-ageing (DSA) in the unaged (WQ) state from 623 to 823 K and it is essentially due to silicon in the solid solution. The degree of DSA decreases with the ageing temperature and DSA does not occur in specimens aged at 973 and 1073 K. In general, the ductility of the WQ as well as the aged material increases with test temperature, except in the range of DSA, where the ductility of WQ material is reduced. The mode of fracture of the WQ specimens remains ductile in the lower and higher ranges of test temperature, but changes to quasi-cleavage at intermediate test temperatures. The minimum in the ductility and quasi-cleavage mode of fracture at 773 K, in the WQ material, is due to strong DSA. Three different modes of fracture, namely faceted, ductile, and mixed intergranular and ductile in the lower, intermediate and higher range of test temperature, respectively, are observed also in the aged conditions (at and above 923 K) of the material. The tensile properties and fracture characteristics in the aged conditions are controlled by the silicides.

1. Introduction

The alloy Ti–6Al–5Zr–0.5Mo–0.25Si (IMI 685) is one of the important members of the near-α-titanium alloys, designed for high-temperature applications in jet engines. It develops different microstructures depending upon the cooling rate, following β-solutioning treatment. Silicon is an important alloying element for creep-strengthening of the alloy. However, precise control of chemical composition and heat treatment is essential to avoid the precipitation of silicon in the form of silicides. The authors have examined the effect of silicide precipitation on the tensile properties and fracture behaviour of the alloy at room temperature, and have shown that precipitation of silicides leads to a drastic impairment of ductility and consequently alters the mode of fracture from ductile to cleavage-like faceting [1]. It may be noted that effective water-quenching of Alloy 685 following β solution-treatment results in martensitic transformation of the β phase, and only a single-phase microstructure with fine platelets of α' results [2]. The ageing of as-water-quenched (WQ) material at elevated temperatures results in the precipitation of silicides [2].

The present work is concerned with effects of ageing treatments at different temperatures from 473 to 1073 K for 24 h, following β-solutioning and water-quenching, on tensile properties and fracture behaviour of Alloy 685, over a range of test temperature from 300 to 823 K.

2. Experimental procedure

The alloy used in the present investigation was obtained from Titanium International, UK, (London) in β-forged and mill-annealed condition as rods of 25 mm diameter. It contained (by wt %) 6.18Al, 5.27Zr, 0.5Mo, 0.28Si, 0.01N, 0.024Fe, 0.14O and 30 ppm H. Tensile specimens with gauge length and diameter of 25 and 5 mm, respectively, were encapsulated in evacuated silica tubes with titanium getter. Following solution-treatment at 1323 K for 0.5 h, they were quenched in water at room temperature following the technique described elsewhere [2]. The WQ specimens were subsequently aged at different temperatures from 473 to 1073 K for 24 h in a vacuum (10^{-5} MPa) quenching furnace. The reported temperatures were controlled to within ± 3 K. The different heat treatments are summarized in Table I. Thin foils for electron microscopy were prepared by the twin-jet electropolishing technique described elsewhere [2].

Tensile tests were conducted in air at different temperatures from 300 to 823 K at a nominal strain rate of $\simeq 1.3 \times 10^{-4}$ sec^{-1} using a servomechanical Instron of 5 ton (5.1 tonne) capacity. Fracture surfaces

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TABLE I The ageing treatments following solutioning at 1323 K for 0.5 h and water-quenching

Heat treatment designation	Ageing treatment	Silicide
WQ	–	Not observed
WQ-A′	473 K, 24 h	Not observed
WQ-A″	673 K, 24 h	Not observed
WQ-A	823 K, 24 h	Not observed
WQ-A1	923 K, 24 h	$S_1 + S_2$
WQ-A2	973 K, 24 h	S_2
WQ-A3	1073 K, 24 h	S_2

were examined using a Philips scanning electron microscope PSEM 500.

3. Results and discussion

3.1. Tensile properties and fracture behaviour at room temperature (300 K)

3.1.1. Tensile properties

The various tensile parameters for the WQ and the specimens aged at different temperatures are recorded in Table II. The influence of the ageing temperature on the yield and tensile strength and percentage reduction in area (RA) at room temperature is shown in Fig. 1. The effect of the ageing treatment is relatively less obvious on the strength parameters as compared to that on the ductility of the material. There is a marginal decrease in the yield strength from ageing at 473 K (Fig. 1). However, the yield strength increases from ageing at higher temperatures, attains a maximum value from ageing at 923 K and starts decreasing with a further rise in the ageing temperature. The marginal decrease in the yield strength from ageing at 473 K could be attributed to recovery of the stresses associated with quenching and a decrease in the density of dislocations in the WQ material. The increase in yield strength from ageing at 673 K and particularly 823 K may be attributed to the formation of clusters of silicon and zirconium atoms in the matrix [3]. The peak value of yield strength from ageing at 923 K may be related essentially to the precipitation of fine silicides. Ageing of the WQ samples [2] at 923 K results in the precipitation of fine S_1 ($(Ti, Zr)_5Si_3$) and S_2 ($(Ti, Zr)_6Si_3$) silicides. The fall in yield strength from ageing at higher temperatures of 973 and 1073 K is due to the precipitation of coarse S_2 silicide with an increased interparticle spacing, and also to less effective solid-solution strengthening because of the considerable depletion of silicon and zirconium from the matrix in the precipitation of the S_2 silicide. The variation of the tensile strength with the ageing temperature was similar to that of the yield strength (Fig. 1). It is obvious from the ratio of tensile to yield strength and also from the value of the work-hardening exponent, n (Table II), that the degree of work hardening is reduced from ageing at 473 K but is increased from that at 673 K. However, there is an almost continuous drop in the degree of work-hardening from ageing at higher temperatures. The lower tensile strength in the WQ-A3 condition than that in WQ (unaged) may be attributed to the lowest work-hardening ability of the material in the former condition (Table II).

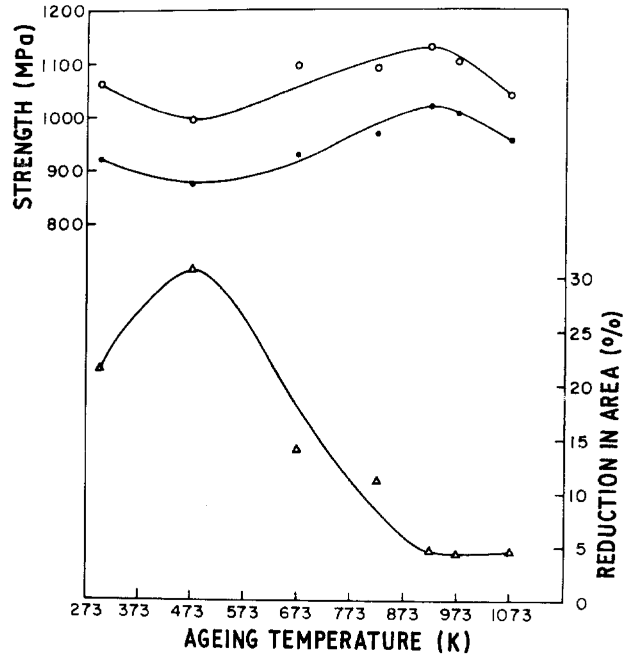


Figure 1 Effect of ageing temperature on strength and ductility at room temperature. (○) UTS, (●) 0.2% yield stress, (△) reduction in area.

The ductility parameters are observed to increase on ageing at 473 K (Table II). This behaviour may be understood in the light of the softening of the material as mentioned above. However, there is a drastic reduction in the ductility parameters; in particular, the reduction in area due to the ageing treatment at higher temperatures, especially at 923 K and above (Fig. 1). It may be seen from Table II that there is relatively less difference between the total and the uniform plastic strain for the WQ-A1, WQ-A2 and WQ-A3 than the other aged conditions, where there is no precipitation of silicides. Thus it is seen that the resistance of the material to deformation beyond plastic instability is reduced due to the precipitation of silicides.

TABLE II Tensile properties in WQ and differently aged conditions, at room temperature

Designation	0.2% yield stress, YS (MPa)	Ultimate tensile strength, (UTS) (MPa)	UTS/YS	RA (%)	Elongation (%)*		n**
					ε_{up}	e_t	
WQ	919	1058	1.151	21.3	4.0	7.2	0.062
WQ-A′	874	995	1.138	30.8	6.2	12.6	0.053
WQ-A″	930	1096	1.178	14.0	4.4	7.3	0.069
WQ-A	966	1090	1.128	11.0	3.5	5.2	0.059
WQ-A1	1020	1132	1.110	4.4	3.8	4.7	0.046
WQ-A2	1005	1102	1.097	4.0	3.6	4.8	0.045
WQ-A3	954	1038	1.088	4.4	3.0	3.8	0.043

*ε_{up} = true uniform plastic elongation, e_t = total elongation
**n = work hardening exponent.

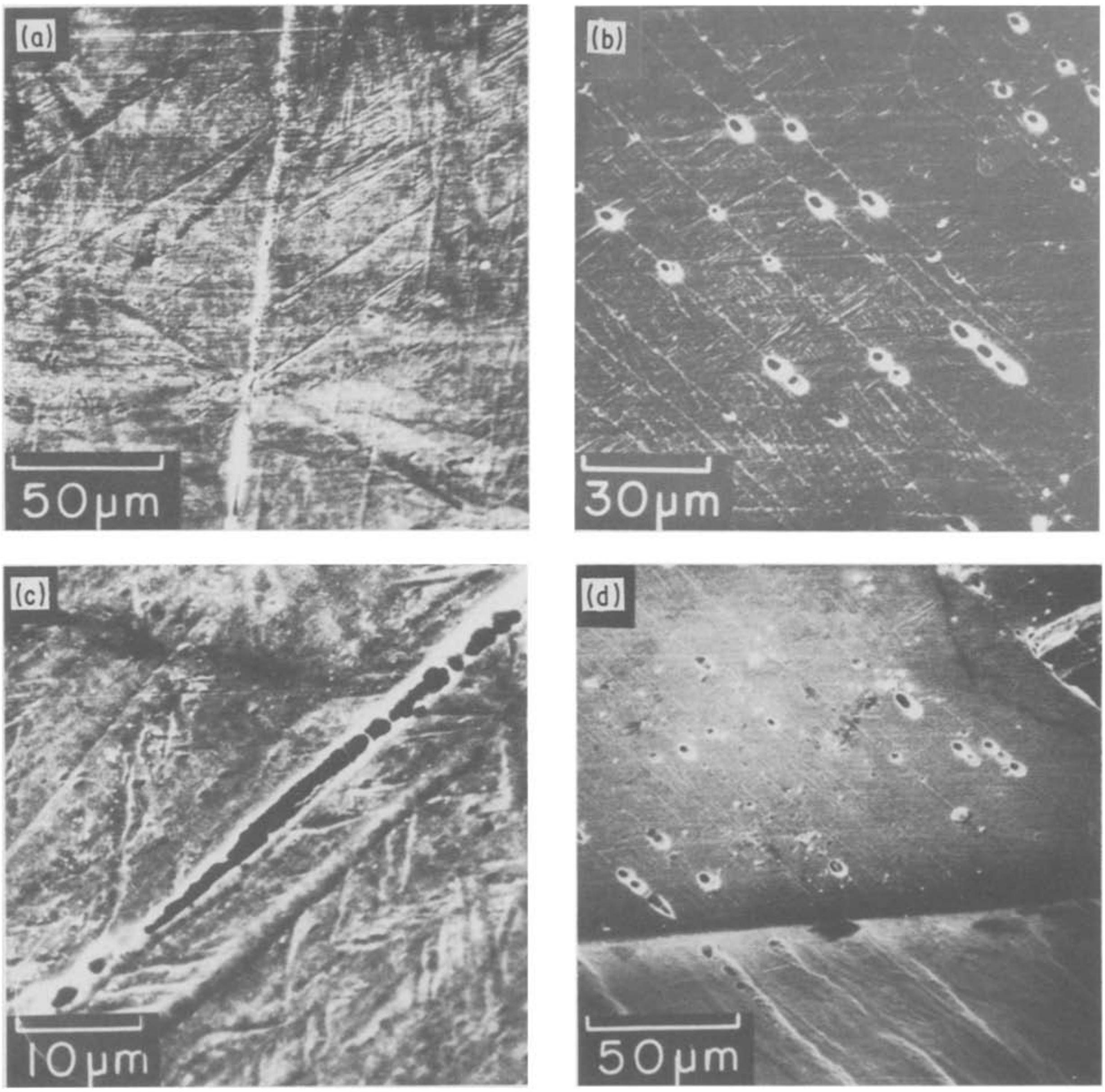


Figure 2 Scanning electron micrographs showing small voids and their linkage in the longitudinal section of Alloy 685 in the WQ-A3 condition tested at room temperature, to give rise to facets on the fracture surface: (a) fine stringer of tiny voids, (b) linkage of small voids along slip traces, (c) formation of a crack through linkage of voids, (d) one-to-one correspondence between the slip bands on the smooth surface and slip traces on the longitudinal section with small voids along them.

The observed differences in the tensile properties of WQ-A1, WQ-A2 and WQ-A3 with respect to WQ may be attributed, mainly, to the effect of silicide precipitates, as ageing of WQ in the temperature range 923 to 1073 K does not lead to any other phase transformation but the precipitation of silicides [2].

3.1.2. Fracture behaviour

The effect of silicide precipitation on the fracture behaviour at room temperature has been described at length elsewhere [1]. The precipitation of silicides promotes planar slip and causes extensive faceting in the fracture surface. The scanning electron micrographs in Fig. 2 give further insight into the process of faceted fracture resulting from the precipitation of silicides. The long stringer of fine voids in Fig. 2a reflects the highly heterogeneous nature of the deformation. Further, it may be noted that these features are from the mid-longitudinal section of the fractured specimen and thus represent the bulk behaviour. It may be inferred from Figs 2b and d that voids form from the interaction of dislocations and silicides in the planes of active slip. The linkage of the tiny voids leads to the formation of a long crack (Fig. 2c). The length of such cracks could be comparable to the diameter of the prior β-grain. The growth of such cracks, through further linkage of small voids lying ahead, results in the formation of the large facets observed on the fracture surface. The faceted fracture and associated embrittling of Alloy 685 in the aged condition may thus be related essentially to the detrimental role of silicide precipitates, as also observed earlier in Alloy 684 [4]. The possibility of embrittlement of Alloy 684, even from Ti_3Al, has been ruled out and attributed only to silicides. However, Blenkinsop *et al.* [5] have attributed the loss in ductility of Alloy 829 to the combined effects, mainly, of the precipitation of silicides and then the compositional changes in the phases.

The WQ-A condition lies between the two extremes,

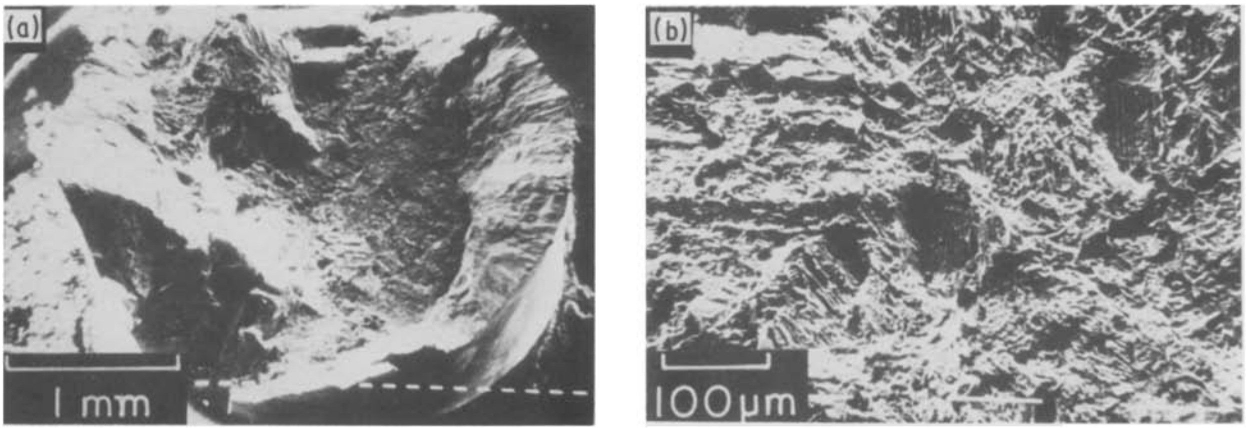


Figure 3 Scanning electron fractographs for the WQ-A condition tested at room temperature, showing (a) fracture surface at low magnification, (b) a mixed mode of fracture consisting of flute-like features on small facets and equiaxed dimples in the central region.

namely WQ (unaged) and WQ-A3 (aged at 1073 K). Its fracture surface (Fig. 3) shows a mixed mode of fracture with dimples and flute-like features which may possibly be related to the clustering of zirconium and silicon atoms in that condition [3].

3.2. Tensile properties and fracture behaviour at elevated temperatures

3.2.1. Tensile properties

Monotonic tensile tests were carried out for WQ, WQ-A, WQ-A1, WQ-A2 and WQ-A3 in the temperature range 300 to 823 K. An important observation made was in regard to prominent serrations in the load–extension curves of the WQ specimens in the temperature range 623 to 823 K, and thus the occurrence of dynamic strain-ageing (DSA). The intensity of serrations decreased and their occurrence shifted to the higher test temperature of 723 K due to pre-ageing of the WQ specimens at 823 K and 923 K. When WQ specimens were aged at the still higher temperatures of 973 and 1073 K, the load–extension curves did not show any serrations in the range of test temperature referred to above.

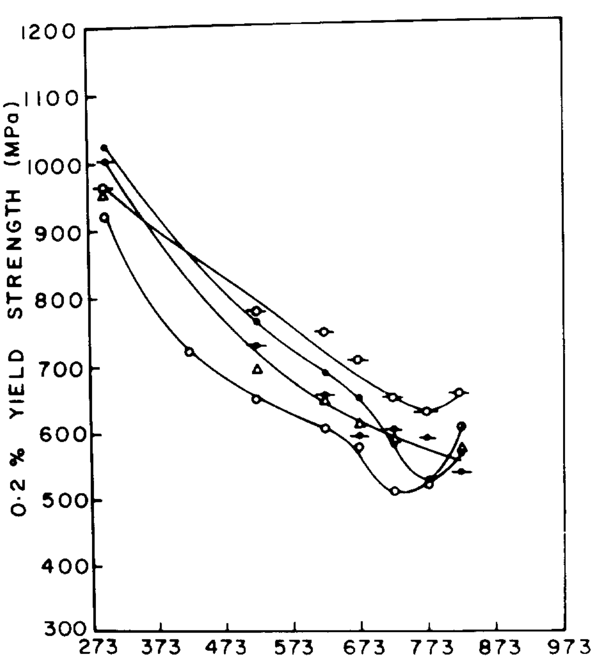


Figure 4 Variation of yield strength with test temperature: (○) WQ, (—○—) WQ-A, (●) WQ-A1, (—●—) WQ-A2, (△) WQ-A3.

The variation of 0.2% yield strength with test temperature for the different heat-treated conditions is shown in Fig. 4. There is a sharp fall in the 0.2% yield strength of WQ between 623 and 700 K, followed by a plateau and peak in the range 700 to 823 K. The dependence of yield strength on test temperature for WQ-A and WQ-A1 conditions is similar to that of WQ condition (Fig. 4). However, DSA occurs in a relatively higher temperature range from 773 to 823 K. The variation of yield strength with temperature is quite different for the WQ-A2 and WQ-A3 conditions, resulting from ageing at higher temperatures of 973 and 1073 K. There is a continuous decrease in the yield strength with test temperature without any plateau or peak in the range of temperature investigated. Similar trends were observed in the variation of tensile strength with test temperature for the respective conditions. Thus it is observed that there is a strong tendency for DSA in the WQ condition, relatively less in the specimens aged at low temperatures (such as WQ-A and WQ-A1), and there is no sign of DSA in the specimens aged at high temperatures, such as WQ-A2 and WQ-A3.

The strong tendency for DSA in the WQ condition may be attributed to the presence of silicon in the solid solution. The absence of DSA in the WQ-A2 and WQ-A3 conditions is obviously due to the depletion of silicon from the solid solution because of extensive precipitation of silicides in these conditions. The smaller extent and higher temperature range of DSA in the WQ-A1 condition could be attributed to partial depletion of silicon from the matrix owing to precipitation of some fine silicides. The lower extent of DSA in the WQ-A condition may be due to the clustering of zirconium and silicon atoms [3] from the ageing at 823 K for 24 h. These observations on the role of silicon in DSA in Alloy 685 are in accord with earlier findings [6–10]. DSA in simpler silicon-bearing titanium alloys has been shown [6–8] to be associated with a strong interaction between silicon and interstitials to cause effective pinning of dislocations. Winstone *et al.* [6] have reported that at any given strain rate and constant interstitial content the temperature of DSA is reduced by a higher amount of silicon in

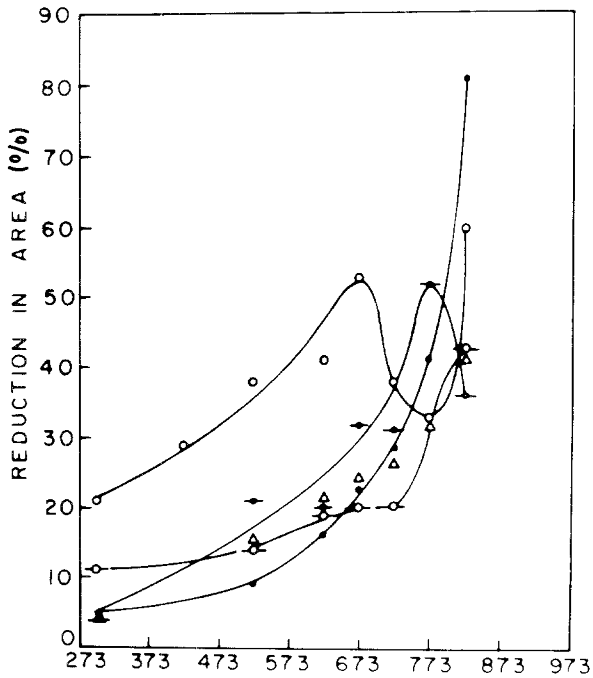


Figure 5 Variation of ductility (RA) with test temperature for (○) WQ, (—○—) WQ-A, (●) WQ-A1, (—●—) WQ-A2, (△) WQ-A3.

solution. Assadi *et al.* [9] have reported DSA in Alloy 685 in the oil-quenched and aged (OQ-A) condition during monotonic deformation in compression in the temperature range 700 to 800 K, and have related it mainly to the presence of silicon in the alloy. The temperature range of DSA in the OQ-A condition [9] is similar to that observed in the WQ-A and WQ-A1 conditions in the present investigation. However, DSA in the WQ condition is observed to occur in a lower range of temperature as compared to the one reported by Assadi *et al.* [9] for the OQ-A condition.

It is obvious from Fig. 4 that in general the strength level of the material is higher for the aged as compared to the unaged condition. The strength at elevated temperatures is observed to be highest for the WQ-A condition, and it may be attributed to a strong interaction of dislocations with pre-existing clusters of solutes and/or probably fine precipitates of silicides at dislocations. The yield strength in the WQ-A1 condition is higher than those in the WQ-A2 and WQ-A3 conditions up to the test temperature of about 673 K, and this may be related to the smaller size of the silicide precipitates in the former condition as compared to the latter ones. However, the strength level of the material in the WQ-A1 condition is observed to be lower than that in the WQ-A2 and WQ-A3 conditions at 773 K, and the strength level of WQ-A1 becomes closer to that of WQ. The strength levels in the WQ-A2 and WQ-A3 conditions are almost comparable.

The variation of ductility with test temperature is shown in Fig. 5. The ductility of the WQ specimens increases with test temperature up to 673 K and is considerably larger than those of the aged specimens

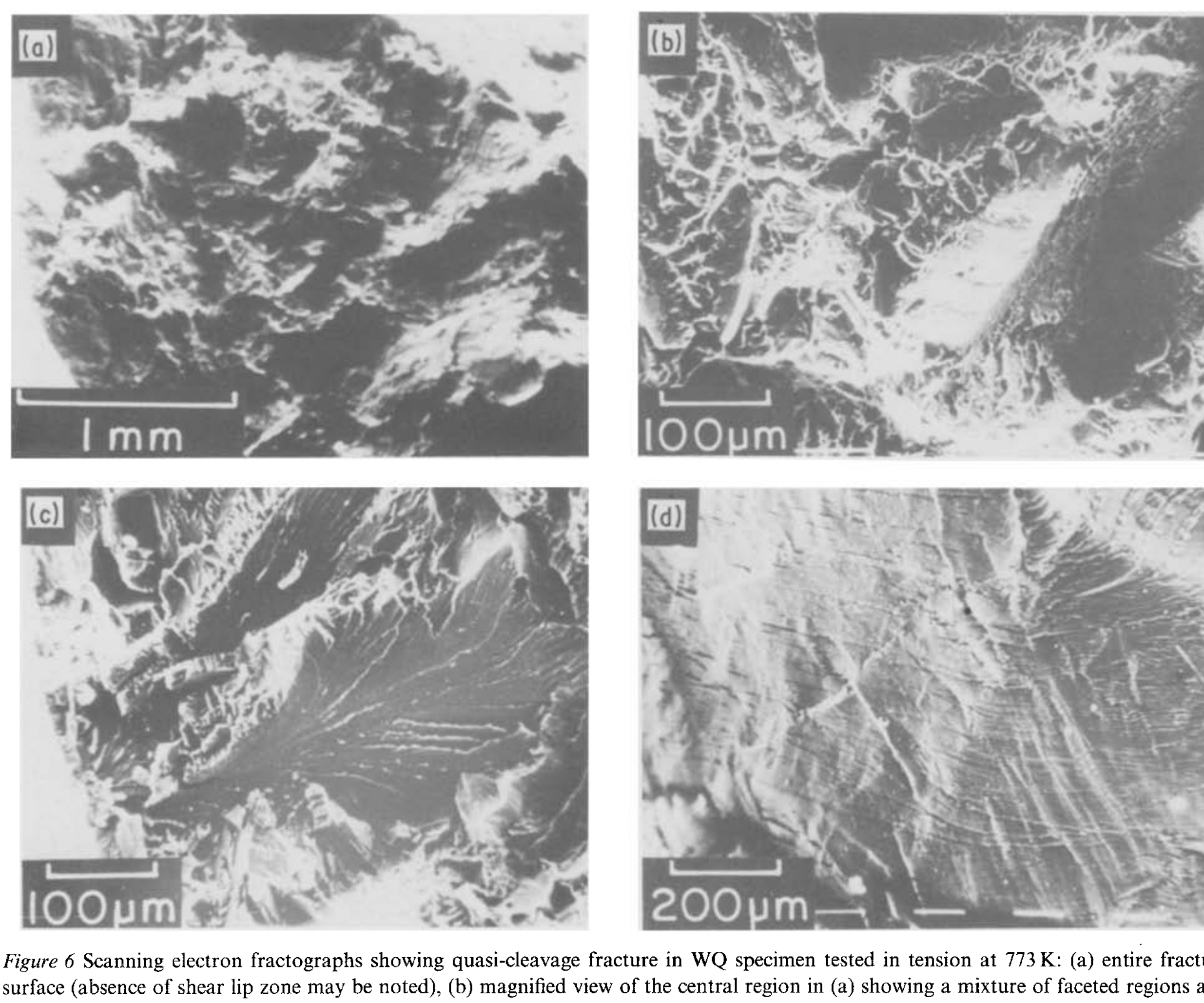


Figure 6 Scanning electron fractographs showing quasi-cleavage fracture in WQ specimen tested in tension at 773 K: (a) entire fracture surface (absence of shear lip zone may be noted), (b) magnified view of the central region in (a) showing a mixture of faceted regions and dimples, (c) cleavage facets with typical river pattern in peripheral region, (d) slip bands and grain boundaries on smooth surface close to the fracture surface.

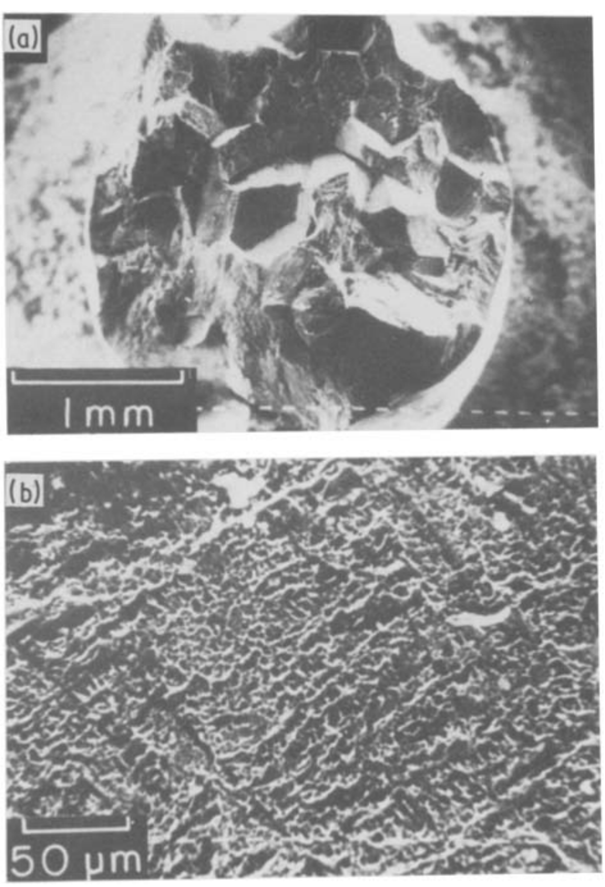
up to that temperature. The ductility of the WQ material is reduced at the higher test temperature of 723 K and reaches a minimum value of 773 K. It may be noted that there was a maximum tendency for DSA at 773 K in the WQ condition. The ductility of the WQ material again increases at 823 K. In general, there is a more or less continuous but slow increase in the ductility of the material aged at different temperatures, up to the test temperature of 673 K. The ductility of the unaged (WQ) and differently aged samples is comparable at the test temperature of 773 K. The variation of ductility with test temperature may be understood from the fracture characteristics of the material in the respective conditions and tested at different temperatures.

3.2.2. Fracture behaviour

In order to bring out the effect of silicides on the fracture characteristics of the material, the fracture behaviour of WQ (unaged) and WQ-A3 (aged at 1073 K for 24 h) will be presented below.

3.2.2.1. WQ condition. The fracture surface of the WQ specimens showed that there were three distinct modes of fracture in the range of test temperature from 300 to 823 K. There was ductile fracture with characteristic dimples from 300 to 523 K.

The mode of fracture was quasi-cleavage from 623 to 773 K (Fig. 6). There is no distinct region of shear lip in Fig. 6a. The quasi-cleavage mode of fracture is quite obvious from Fig. 6b, which shows a mixture of cleavage facets and small dimples in the central region of the fracture surface. Distinct cleavage facets with river patterns may be seen in the peripheral region (Fig. 6c). The strengthening from DSA and the consequent reduction in the ductility lead to cleavage faceting in the material. The low ductility of the WQ material at 773 K (Fig. 6) may thus be understood from the quasi-cleavage mode of fracture at that temperature.



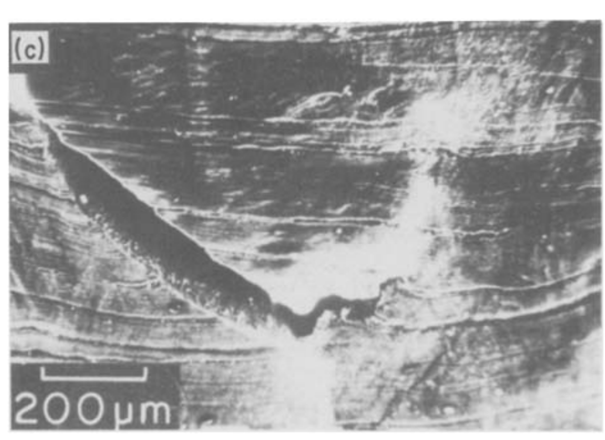
The mode of fracture is ductile at the higher test temperature of 823 K, and this is quite consistent with the observed increase in ductility at this temperature. Though the fracture both at 523 and 823 K was ductile in nature, the ductility was higher at 823 K than at 523 K and this is related to the more homogeneous deformation at the higher tést temperature.

3.2.2.2. WQ-A3 condition. Three distinct ranges of temperature, with specific modes of fracture, are identified also for the WQ-A3 condition. Faceted fracture was observed in the temperature range 300 to 623 K. The characteristics of the faceted fracture are described elsewhere [1].

The fracture was ductile in nature from 623 to 723 K. The occurrence of ductile fracture may be related to a more homogeneous deformation and thus a less detrimental role of silicides at the interplatelet boundaries of martensitic α' at elevated temperatures. The high ductility in that range of temperature (Fig. 5) may thus be understood in the light of a more homogeneous deformation and ductile fracture.

The fracture behaviour was quite different at the still higher temperature of 823 K (Fig. 7). There are intergranular and transgranular facets. The intergranular facets reveal fine and shallow dimples at high magnification (Fig. 7b), and these features are similar to those reported by Hall and Hammond [11] in Alloy 685 in the furnace-cooled condition which failed through intergranular cracking. The tendency for intergranular cracking becomes quite evident from Fig. 7c. This mode of fracture is in accord with the poor ductility (Fig. 5) at this temperature. The occurrence of intergranular fracture may be due to the precipitation of silicides along the grain boundaries (Fig. 8).

Figure 7 Scanning electron fractographs showing intergranular and transgranular facets in WQ-A3 specimen tested at 823 K: (a) mixture of intergranular and transgranular facets, (b) enlarged view of the intergranular facet in (a) showing fine dimples on the facet, (c) large intergranular crack and slip bands on smooth surface close to the fracture end.



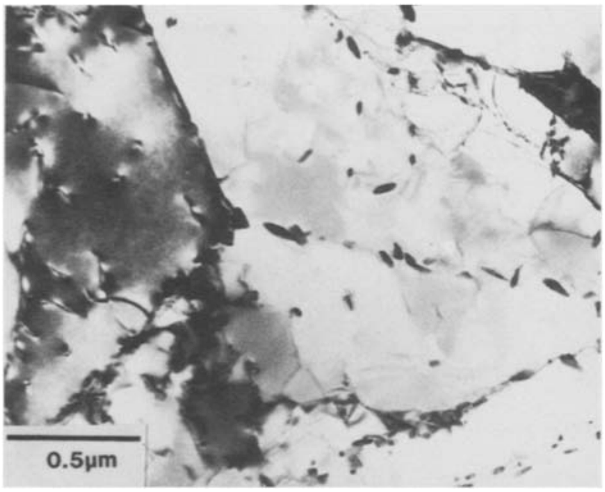


Figure 8 Transmission electron micrograph showing precipitation of silicides along grain boundaries in the WQ-A2 condition.

4. Conclusions

The following conclusions are drawn from the present investigation.

1. Precipitation of silicides in Alloy 685 causes marginal strengthening but leads to a drastic reduction in the ductility at test temperatures from 300 to 673 K. The detrimental effect of silicides on ductility is less at higher test temperatures, above 673 K.

2. There is a strong tendency for DSA in the WQ condition from 623 to 823 K due to the presence of silicon in the solid solution. DSA is absent in the WQ-A2 and WQ-A3 conditions as silicon is depleted from the matrix and precipitated out as silicides.

3. The mode of fracture of the WQ material is ductile in the lower and higher ranges of test temperature but quasi-cleavage in the temperature range of DSA.

4. There are three different modes of fracture, namely faceted cleavage, ductile, and mixed intergranular and ductile in the lower, intermediate and higher ranges of test temperature, respectively, for the WQ-A3 material.

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