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On Silicides in High Temperature Titanium Alloys

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Abstract. High temperature titanium alloys like IMI 685 contain small amounts of silicon ($\approx$ 0.25 wt. per cent) to improve creep resistance. Different types of silicides, namely Ti_5Si_3, $(TiZr)_5Si_3$ (S_1) and $(TiZr)_6\,Si_3$ (S_2), have been observed to precipitate in various silicon-bearing titanium alloys depending upon their composition and heat treatment. The precipitation of silicides, their orientation relationship with the matrix in different alloys, and the beneficial influence of thermo-mechanical treatment on the distribution of silicides have been pointed out. The effect of silicides on mechanical properties and fracture of the commercial alloy IMI 685 is also indicated.

1. Introduction

Several near α titanium alloys have been designed for elevated temperature applications upto 823 K. It is now established[1-9] that small amounts of silicon in the titanium alloys lead to significant improvement in their creep resistance. There have been differing views about the precise mechanism of creep strengthening due to silicon. While some investigators attribute it to solid solution strengthening through dynamic strain aging[5,8] (DSA), the others relate it to pinning of mobile dislocations by silicide precipitates' during creep. Different types of silicides[10-14] precipitate depending upon composition of the alloys and heat treatment. The type, size and distribution of silicides is expected to have an effect on mechanical properties of these alloys. However, only limited investigations have been carried out so far on these aspects[23]. The purpose of this article is to present the work related to silicide precipitation and effect on mechanical properties in certain high temperature titanium alloys like IMI 685.

2. Silicides in α and Near-u Titanium Alloys

Different types of silicides identified in various silicon bearing titanium alloys subjected to different heat treatments are presented in a summary form in Table 1

2. I *α-Alloys*

The aging of the binary *Ti-Si* alloys'“, following solution treatment and water quenching, results in precipitation of Ti_5Si_3 silicides. The silicides precipitate heterogeneously on dislocations and grow in the form of rods in < I 120> α directions'“. Alloying elements, particularly *Zr*, *Al*, *Sn* and *Mo* have been found to have considerable effect on the characteristics and precipitation kinetics of silicides in titanium alloys. In the ternary alloy *Ti-5Zr-1Si*, zirconium has been reported" to reduce the solubility of silicon in titanium. Part of the added zirconium is incorporated in the resulting complex silicides[10’12], namely S, $[(TiZr)_5 Si_3)]$ and $S_2[(TiZr)_6 Si_3]$. The presence of zirconium in the ternary alloys *Ti-Zr-Si* obviates the matrix nucleation of silicide precipitates by Guinier-Preston (GP) zone formation. However, zone formation does not occur in the ternary *Ti-Zr-Si* alloys when the amounts of zirconium and silicon are less than the critical values of 5 wt. per cent and 1 wt. per cent respectively. Thus, actual amounts of zirconium and silicon in the ternary alloys have marked influence on G.P. zone solvus. Further, zirconium also reduces the rate of growth of the silicide precipitates.

In the quaternary alloy *Ti-Al-Zr-Si*, aluminium is observed'* to cause drastic reduction in the rate of silicide precipitation and hence longer deviations are required for the precipitation of silicides in these alloys as seen in Table 1. However, it is important to mention that the sequence of precipitation as well as the nature of the final equilibrium precipitates is not affected by the presence of aluminium and they remain essentially the same as in the ternary alloy *Ti-Zr-Si*. Crossley and Turner[15] have found that the solubility of silicon in titanium is insensitive to the aluminum content. This observation has been further strengthened by the findings of Flower et al[12]. Further, it has been shown by Antony[10] that aluminium is not incorporated into the silicide precipitates. The Pseudo-binary phase diagram established by Antony[10] for *(Ti-6Al-3Sn-5Zr)-Si* system shows that the alloying elements (Al-Zr-Sn) raise the eutectoid reaction temperature and lower the terminal solubility of silicon in titanium. The reduction in terminal solubility of silicon is essentially due to zirconium.

2.2 *Near-u Alloys*

In the commercial near a alloys like IMI 685, silicon content is limited to a maximum value of about 0.25 wt. per cent owing to considerable reduction in its solid solubility due to presence of the other alloying elements. In view of higher aluminium and lower silicon content in the near α alloys, silicide precipitation is expected to be much slower than that in the silicon bearing α alloys mentioned above.

There are two important factors, namely the cooling rate following solution treatment and the aging temperature, which affect silicide precipitation in the near a alloys. Generally silicides are not observed to precipitate in the fast cooled specimens. Slow cooling (< 0.1°Cs^{-1}) following soaking in the β phase field has been shown to cause precipitation of the equilibrium silicide S_2 in alloy IMI 685[13]. Such silicide precipitation in this alloy has been observed to occur in the retained β phase, or at the α/β interface or in the a phase in the vicinity of the α/β interface or inside the a platelets. Banerjee et al.[16] suggest that the precipitation on cooling occurs primarily in the β phase and any silicides observed within the a phase in the vicinity of α/β interfaces have been incorporated there by the migration of the α/β interface around the silicide as the α phase grows and consumes the β matrix.

The aging temperature has a significant effect on silicide precipitation in the a and the near α alloys. The commercial aging treatment at 823 K for 24 hrs does not lead to any resolvable silicide precipitation in the water quenched (WQ), oil quenched (OQ) or even in the furnace cooled (FC) specimens of alloy 685. It is evident from the transmission electron micrograph as shown in Fig. I, for the furnace cooled and aged condition (823 K-24 hrs) of alloy 685, that there is no trace of any resolvable silicide. However, aging at temperatures higher than 823 K leads to silicide precipitation. Both types of complex silicides S_1 and S,, referred to earlier, are observed[15] to precipitate on aging at 923 K for 24 hrs. Aging at still higher temperatures in the range 973-1073 K results in precipitation of only the S_2 silicide. The S_2 silicide may be considered to be the equilibrium silicide in alloy 685 since only this silicide results on very slow cooling of the alloy as also observed by Barbier, et al.[13] The transmission electron micrograph

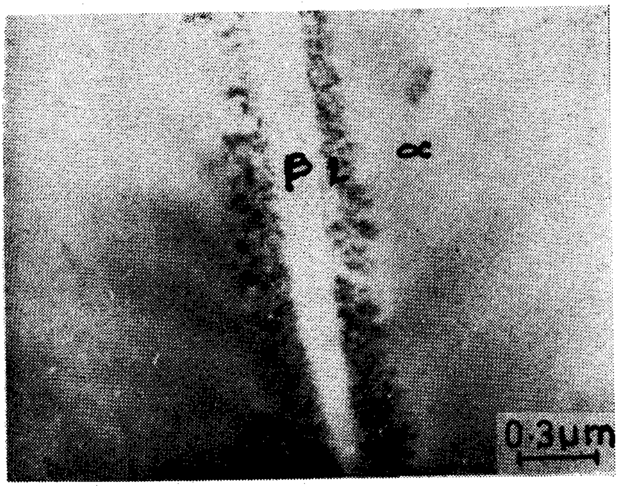

Figure 1. Transmission electron micrograph showing absence of silicide precipitates in FC-A condition in alloy IMI 685.

Table 1. Silicides in α and near α titanium alloys

S. No.	Alloy	Heat treatment: Solution treatment	Heat treatment: Aging	Heat treatment designation	Type of silicide	Preferred sites for precipitation	Shape and size	Reference
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)
1	*Ti-Si*	1473 K-24 h water quench	823 K-2 to 400 h	—	Ti_5-Si_3	Dislocations and martensite plate boundaries; heterogeneous precipitation	Rod	12
2	*Ti-5Zr-1Si*	1473 K-24 h water quench	823 K-17 h	—	Unresolvable	—	—	12
			823 K-300 h	—	S_1	Dislocations; homogeneous precipitation	Discs or short rods of 25 nm	12
			923 K-5 h	—	S_1 & S_2			
			1073 K-150 h	—	S_1 & S_2	Heterogeneous precipitation	500 nm	12 12
3	*Ti-5Zr-5Al-1Si*	1473 K-24 h water quench	823 K-833 h	—	1st zones visible	—	—	12
			823 K-1667 h	—	S_1	—	—	12
			923 K-170 h	—	—	Heterogeneous precipitation	35 nm in diameter	12
4	*Ti-6Al-3Sn-5Zr-0.1* to *1.0Si*	Annealed at 1143 K to 1303 K	—	—	S_1	—	—	10
5	IMI 685 (*Ti-6Al-5Zr-0.5Mo-0.25Si*)	1473 K-24 h water quench	823 K-24 h	WQ-A	Unresolvable		—	12
			823 K-168 h	—	—	A few at grain boundaries	—	12

contd.

Table I Contd.

(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)
		1323 K-1/2 h water quench	923 K-24 h	WQ-A_1	S_1 & S_2	Interplatelet boundaries of α′	50 nm ellipsoidal	14
			973 K-24 h	WQ-A_2	S_2	Platelet boundaries of α′	100 nm	14
			1073 K-24 h	WQ-A_3	S_2	—	250 nm	14
		1323 K-1/2 h oil quench	823 K-24 h	OQ-A	Unresolvable	—	—	14
		As received (forged & mill annealed)	—	AR	Unresolvable	—	—	14
		1323 K-1/2 h furnace cool	823 K-24 h	FC-A	Unresolvable	—	—	14
		1323 K-1 h slow cool ($<0.1°C\ s^{-1}$)	—	SC	S_2	In β phase, across interface phase, inside α phase	Trapezoidal precipitates 100 nm at $0.08°C\ s^{-1}$ 2000 nm at $0.003°C\ s^{-1}$	13
6	*VT-9* (*Ti-6.5Al-3.5Mo-2Zr-0.25Si*)	1323 K-1/2 h water quench	923 K-24 h	WQ-A_1	S_2	Heterogeneous precipitation	Ellipsoidal	
			1073 K-24 h	WQ-An	S_2			
7	*Ti-5Al-5Sn-2Zr-0.8Mo-0.7Si*	As received	—	—	S_1	Interplatelet boundaries	Spherical 300 nm	11
8	*Ti-4.5Al-2Sn-3Ga-3Mo-0.5Si*	1298 K-1 h air cool	868 K-4 h	—	S_1	Interplatelet boundaries	75-150 nm	11
9	*Ti-6Al-5Zr-1W-0.4Si* (IMI 684)	1323 K-1 h oil quench	773 K-300 h	—	S_1	Predominantly along the martensite platelet	Elliptical	6

contd.

Table 1. (contd.)

(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)
10	Ti-5.5Al-3.5Sn-3Zr-1Nb-0.25Mo-0.3Si (IMI 829)	1323 K-1 h oil quench	898 K-24 h 848 K to 1073 K	— —	$(TiZr)Si$ $(TiZr)Si$	Predominantly along the α lath boundaries	Elliptical	22
		1248 K-1 h air cool		—	S_2	Predominantly along the interface phase and in β	Elliptical	17
		1248 K-1 h oil quench	—	—	$(TiZr)Si$	Predominantly along retained β	Elliptical	17
		1323 K-1/2 h water quench	898 K-24 h	—	s & S_2	Interplatelet boundaries	Elliptical	17
		1323 K-1/2 h water quench	473 K-24 h	—	S_2	Interplatelet boundaries	Elliptical	17

S_1 : $(TiZr)_5Si$ (hexagonal crystal structure; a_0 = 0.773 nm, c_0 = 0.532 nm)
S_2 : $(TiZr)_6Si$ (hexagonal crystal structure; a_0 = 0.70 nm, c_0 = 0.368 nm)

in Fig. 2 shows silicide precipitation **predominently** along the boundaries of the α^1 platelets in alloy 685 in the WQ-A3 condition. Similar observations on the preferential precipitation of silicides along the a' platelet boundaries of α^1 were also made in the other aged specimens like W Q-A2.

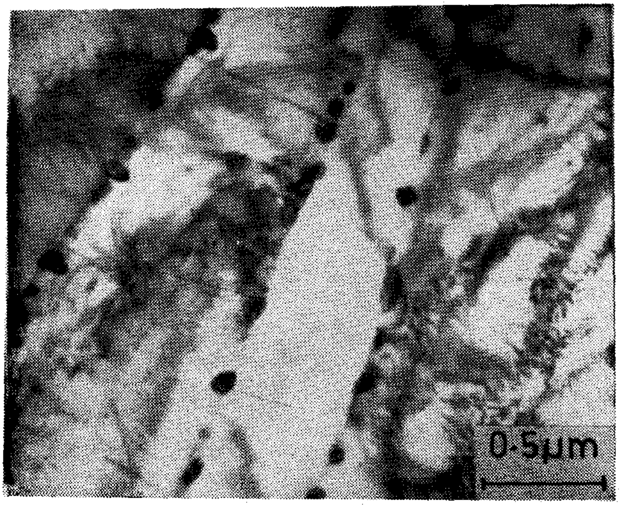

Figure 2. Transmission electron micrograph showing coarse silicide precipitates predominantly at the platelet interfaces in WQ-A3 condition-Alloy IMI 685.

Sridhar et al[17] have presently undertaken a study of silicide precipitation in an IMI alloy (829) designed for a temperature capability better than IMI 685. Thdir observation of elliptical silicide particles in IMI 829 in the as oil-quenched or air-cooled condition, following soaking at 1248 K (alpha + beta range) is noteworthy. They do not, however, find silicides on continuous cooling from the beta phase (e.g. 1323 K.). Beta treated IMI 829 alloy yielded silicide precipitates (Fig.3) when aging was carried out after solutionising and cooling (Table I) which is in line with the observations in IMT *685.*

The examination of the water quenched specimens of another important a + β alloy VT-9, aged in the temperature range 923-1073 K, **revealed**[18] precipitation of only the S_2 silicide. 'The diffraction patterns from the S_2 silicide in alloy *VT-9* are shown in Fig. 4.

It is obvious from the data in Table I that in ternary *(Ti-Zr-Si)* and quaternary *(Ti-Al-Zr-Si)* titanium alloys silicide S_1 precipitates at lower temperatures of aging and both S_1 and S_2 precipitate at higher temperatures of aging. However, when small amount of molybdenum is also present as in alloy IMI 685, both S_1 and S_2 precipitate

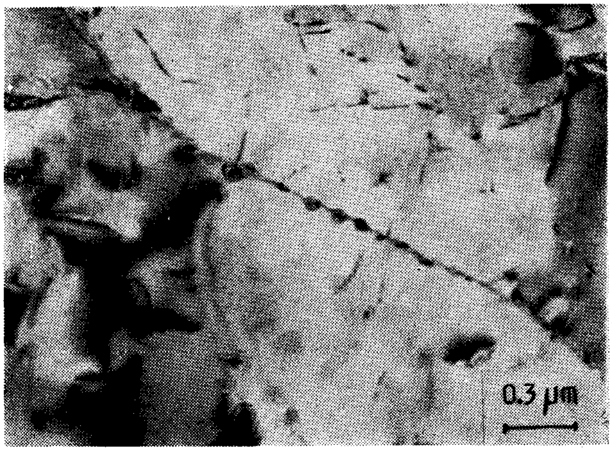

Figure 3. Alloy IMI 829 : Transmission electron micrograph showing silicide precipitates on aging at 898 K for 24 h, after solution treating in β at 1273 K for 1 hr followed by water quenching (Sridhar, et al[17]).

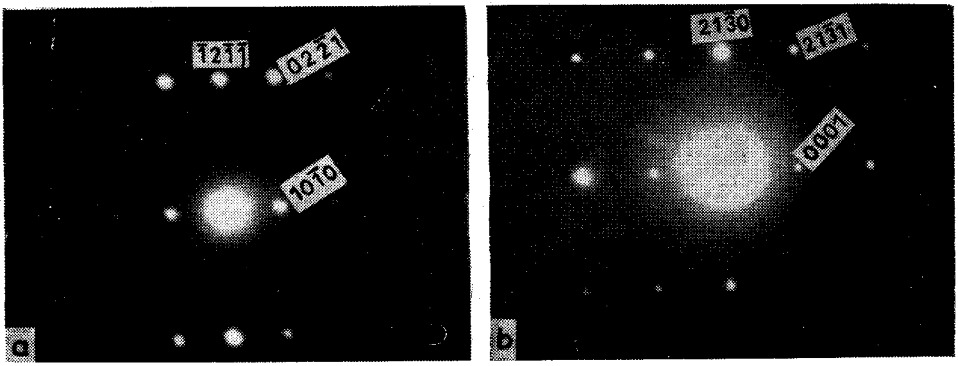

Figure 4. Seleeted area diffraction patterns from the S_2 silicide phase in alloy VT-9 with (a) $B = [\bar{1}2\bar{1}6]$ and (b) $B = [4\bar{5}10]$.

at lower temperature of aging at 923 K and only S_2 silicide is observed to be stable at higher temperatures of aging up to 1073 K. When there is higher content of molybdenum, as in alloy VT-9, only the S_2 silicide is observed to precipitate even at the lower temperature of aging at 923 K and the same one is found also at the higher temperature of aging upto 1073 K. Thus, molybdenum while not incorporated in the silicide precipitates, appears to have significant effect on the structure of silicides. A consideration of all these results suggests[14] that the kinetics of silicide precipitation in the β phase is far more rapid than in α and that the prexisting dislocation substructure in a may exert a strong influence on the silicide distribution.

3. Orientation Relationship Between the Matrix and the Silicides

In the binary *Ti-Si* alloys, the orientation relationships between α^1 and the hexagonal silicide Ti_5Si_3 have been established’* as

$(01\ \bar{1}0)\ \alpha^1//\qquad (01\bar{1}0)\ Ti_5Si_3$

$(2\bar{1}\ \text{io})\ \alpha^1//(2\bar{1}\ \text{ii})\ Ti_5Si_3$

Flower et al[12] have shown the predominant orientation relationships between the matrix and the silicide S, in the ternary alloy *Ti-5Zr-0.5Si* as

$(0001)\ \alpha^1//(0001)\ S_1$

$(01\bar{1}0)\ \alpha^1//(21\bar{3}0)\ S_1$

They have also found that the same orientation relationship exists even in the quaternary alloy *Ti-Al-SZr-l.OSi.*

Ramachandra and Singh[19] have established the orientation relationships between the matrix and the silicide S_2 in alloy IMI 685 in the water quenched and aged condition as follows :

$(10\bar{1}0)\ a'//(1\bar{2}1\ I)\ S_2$

$[0001]\ \chi^1//(\bar{1}2\bar{1}6)\ S_2$ OR $[1\bar{2}10]\ \alpha^1//[\bar{1}010]\ S_2$

However, in the same alloy 685, in the slow cooled condition, Barbier et al[13] have found a different orientation relationship between the three co-existing phases α, β and the silicide S_2 indicated below :

$[1\bar{2}30]\ S_2//[110]_\beta//[\bar{1}011]_\alpha$

$[0001]\ S_2//<00\bar{1}>//<0\bar{1}1\ 1>$

The disparity in the orientation relationships established in the two investigations has been attributed to an error[19] in the interpretation of the diffraction patterns in the work of Barbier et al[13].

4. Effect of Thermomechanical Treatment on Silicides

From the information assembled in Table 1, it is clear that in general silicide precipitation is heterogeneous in the thermally treated titanium alloys. It is desirable to have a more homgeneous distribution of silicides for better mechanical properties of these alloys. In alloy 685 it has been observed that cold working of the water quenched specimen prior to aging at 973 K leads to a more homogeneous precipitation of silicides in the matrix (Fig. 5a) than in the material not subjected to cold working prior to the aging treatment (Fig. 5b). However, no investigations have been made on the effect of cold working on mechanical properties of the near α titanium alloys. Such investigations are desirable.

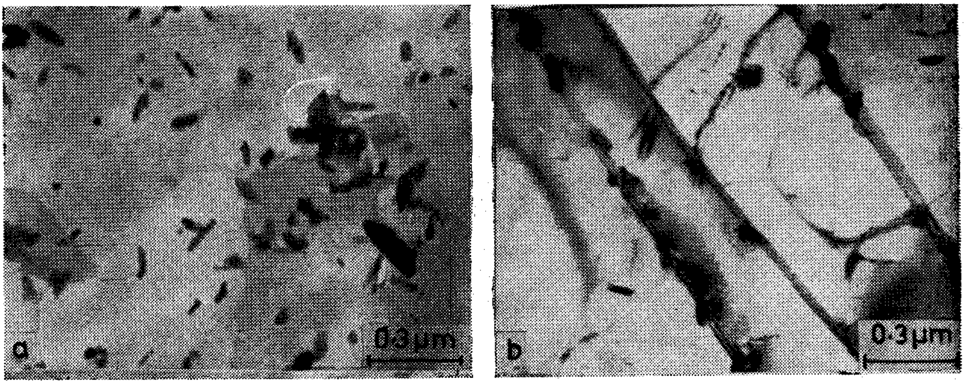

Figure 5. Transmission electron micrographs showing (a) a more homogeneous distribution of silicides in thermomcchanically treated specimen and (b) silicide precipitation predominantly along the interplatelet boundaries in thermally treated (WQ-A2) specimen-Alloy IMI 685.

Tien et al[20] have shown that ductility of a titanium alloy increases by change of the silicide morphology from Widmanstatten plates to evenly distributed cuboids of the silicide precipitates.

5. Effect of Silicides on Mechanical Properties of Titanium Alloys

5.1 *Creep Behaviour*

Assadi et al[8] have studied the creep resistance of certain alloys of the *Ti-Al-Zr-Mo-Si* system in the temperature range 660 to 860 K. They have observed that prolonged aging of the alloys at 923 K prior to creep testing reduces the creep strength. Stress-assisted precipitation of silicide particles and/or E-phase on dislocations was observed to occur during the creep test itself in the elevated temperature range. They have suggested that creep deformation is controlled by the rate at which the dislocations can break away from these precipitates. However, in general they have concluded that precipitation occurring even during the creep test leads to weakening of the alloys.

Paton and Mahoney' have also studied creep behaviour of some silicon bearing titanium alloys at 811 K. They have found that creep resistance of the *Ti-5Zr-0.5Si* and *Ti-5-Al5-Zr0.5Si* alloys is highest for the solution treated condition and lowest for the material with uniform silicides in the microstructure. The creep resistance is intermediate between the two limits for the material with grain boundary silicides. Electron microscopy of the creep tested specimens revealed that dislocations were decorated with precipitates, which they have presumed to be silicides of the type $(TiZr)_5Si_3$.

More recently Mukharjee et al[21] working with the alloy *VT-9* have found that silicon is retained in α solution when cooled at a rate faster than 0.1°C/sec from the β phase, but precipitates during cooling at slower cooling rates, essentially within the β or at the α/β interface. They also report that creep strain is affected by silicon only in samples cooled at the faster rate. This observation was possible when work was carried out with two alloys, (I) VT-9 with nominal composition and (2) *Si*-free *VT*-9. However, more or less similar creep strains were reported for both alloys for cooling rates below 0. 1°C/sec. Transmission electron microscopy did not reveal any silicides on dislocations in crept samples of *VT*-9. This is probably because the alloy VT-9 has lower zirconium content than IMI 685.

5.2 Tensile Properties and Fracture

The effect of silicide precipitation on tensile properties at room temperature and fracture behaviour of the alloy 585 have been investigated by Ramachandra and Singh.[22] The tensile properties in the unaged (WQ) and aged (WQ-A3) conditions are recorded in Table 2. It is seen that there is relatively less effect of the aging treatment on the strength value as compared to that on the ductility parameters, particularly the reduction in area. The marginal improvement in the yield strength of the aged specimen is because of nominal difference between the strengthening caused by silicide particles and the loss of solution strengthening due to depletion of silicon and zirconium in the matrix as a result of precipitation of silicide particles. The lower ultimate tensile strength in the aged condition may be attributed to lower work hardening capacity of the material as indicated by the low value of the work hardening exponent. The drastic reduction in the ductility of the alloy in the aged condition is essentially due

Table 2. Tensile parameters for alloy 685 in WQ and WQ-A3 conditions
(Average of three test results)

Heat treatment designation	0.2% Y.S (MPa)	UTS (MPa)	Reduction in area (%)	Elongation (%)		Work hardening exponent n
				Uniform plastic elongation (u_p)	Total elongation (e_f)	
WQ	919	1058	21	4	7.2	0.062
WQ-A3	954	1038	4	3	3.8	0.045

to silicide precipitates which are observed to change the mode of fracture from ductile to quasi-cleavage as discussed below.

The fractographs in Fig. 6 bring out the distinct differences in the fracture behaviour of the alloy 685 in the unaged and aged conditions. While there are usual fibrous and shear lip zones in the fracture surface of the unaged specimen (Fig. 6a), in sharp contrast, there are facets of varying sizes and smoothness in the fracture surface of the aged specimen (Fig. 6b). The examination of the fracture surfaces at higher magnifications reveals that while there are equiaxed dimples in the fibrous zone of the unaged specimen (Fig. 6c), there are flute like features in case of the aged specimen (Fig. 6d). Since the ductile fracture resulting due to nucleation and growth of voids is known to involve high energy, the observed higher ductility of the material in the unaged condition is understandable.

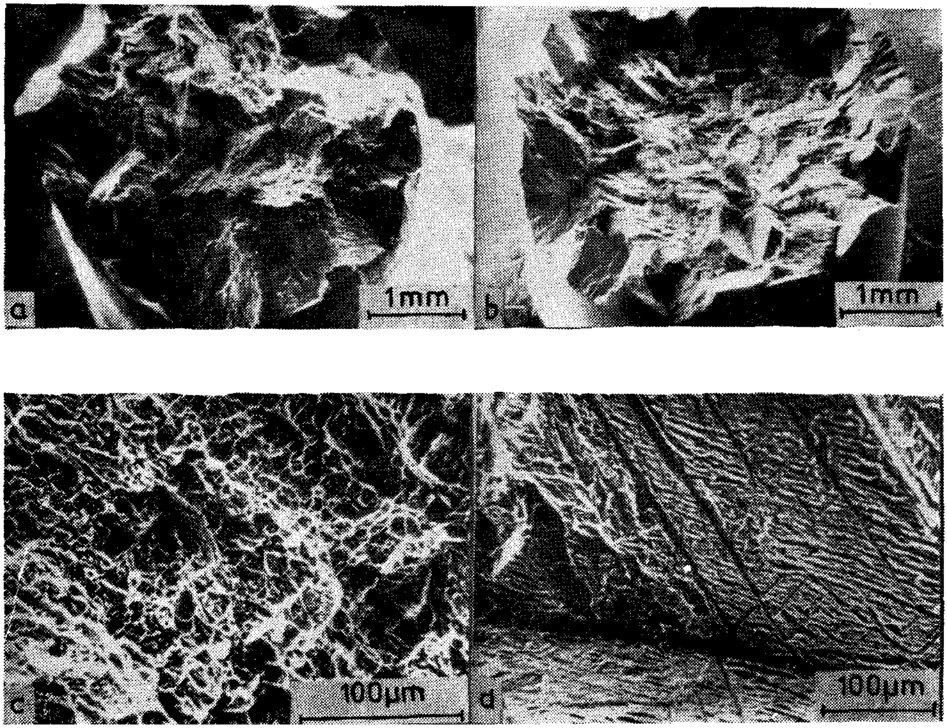

Figure 6. Alloy IMI 685 : Scanning electron fractographs showing (a) and (b) fracture surfaces of unaged and aged specimens respectively, (c) and (d) equiaxed dimples and fluted features in the central regions of the fracture surfaces of the unaged and aged specimens respectively.

The presence of the flutes in the fracture surface of the aged specimen may be taken as indicative of highly localised shear deformation. The increased tendency for intenes shear type of deformation in the aged condition has further been supported by the occurrence of parallel stringers of voids in longitudinal sections of the aged specimen. Thus we believe the observed poor ductility and faceted fracture in alloy 685 in the water quenched and aged condition is due to the precipitation of silicides which promote the tendency for heterogeneous slip. While these observations pertain to aging at temperatures well above those normally used in the commercial heat-treatment of the alloys, similar losses in ductility could in principle be induced by melt and processing induced siliconfsilicide segregation, and therefore the effect is important and must be noted.

6. Conclusion

Different types of silicides precipitate in various silicon bearing titanium alloys like IMI 685 depending upon the composition of the alloys and its heat treatment. The silicides have definite orientation relationships with the matrix. Thermomechanical treatment promotes the tendency for homogeneous distribution of the silicides. In general, the precipitation of silicides leads to reduction in room temperature tensile ductility and creep resistance at elevated temperatures.

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