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Quantitative fractography of SiC whisker–Si_3N_4 matrix composites

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An SEM quantitative stereophotogrammetry technique was developed and employed to analyse the fracture surfaces of VLS SiC whisker–Si_3N_4 matrix composites. This technique has quantitatively established that increased surface roughness is associated with increased fracture toughness for these composites. Matrix grain morphology and whisker/matrix interfacial characteristics are contributing factors to composite surface roughness.

1. Introduction

Significant toughening has been observed in Si_3N_4 composites reinforced with vapour–liquid–solid (VLS) SiC whiskers [1]. However, the mechanisms responsible for this observed toughening are not well established. Two possible toughening mechanisms are crack deflection [2] and crack bridging [3].

The application of quantitative fractography may help to shed light on the fracture mechanisms operative in whisker-reinforced composites. In essence, this approach seeks to quantify aspects of the fracture surface. Modern approaches have recently been reviewed by Banerji [4]. The two current preferred approaches are vertical sectioning procedures and stereophotogrammetry.

The purpose of the present investigation was to develop a scanning electron microscopy (SEM) stereophotogrammetry technique for the quantitative measurement of fracture surface roughness, and then apply this technique for the quantification of fracture surfaces obtained in VLS SiC whisker–Si_3N_4 matrix composites.

2. Experimental procedure

2.1. VLS SiC whisker–Si_3N_4 matrix composites

The composites examined were those described previously [1], and detailed fabrication conditions and mechanical property measurements may be found therein. In summary, these composites were fabricated by dry blending VLS SiC whiskers and Si_3N_4 powders (MgO densification aid employed), then consolidating by hot pressing at temperatures of 1600, 1750 and 1850 °C.

Chevron-notched bend fracture toughness was measured as a function of vol % SiC whiskers and hot-pressing temperature [1]. These results are shown in Fig. 1. Toughness increased with increasing volume per cent of whiskers, and increasing hot-pressing temperature.

For the present investigation, the chevron-notched bend specimen fracture surfaces of four of the composites were examined. The details of these specimens are given in Table I. Basically, a set of specimens was

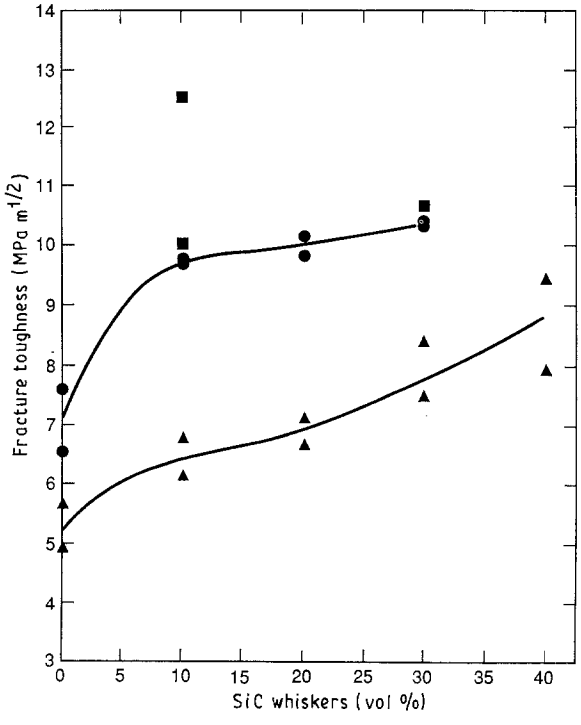

Figure 1 Composite chevron-notched bend fracture toughness as a function of volume % SiC whiskers and hot-pressing temperature. From [1]. Hot pressed at: (●) 1750 °C, (■) 1850 °C, (▲) 1600 °C.

TABLE I Details of specimens examined

Hot-pressing temperature (°C)	SiC whiskers (vol %)	Hot-pressed density (% theoretical)
1600	10	99
1750	10	99
1850	10	99
1600	40	91

examined at constant 10 vol % SiC whisker loading, for hot-pressing temperatures of 1600, 1750 and 1850 °C, to investigate effects of hot-pressing temperature on fracture surface characteristics. Specimens were also studied at constant 1600 °C hot-pressing temperature, for whisker loadings of 10 and 40 vol %, to investigate effects of whisker volume fraction. Pure Si_3N_4 matrix fracture surfaces were essentially flat for all hot-pressing temperatures in comparison to the composites and, hence, were not analysed in detail.

2.2. SEM quantitative stereophotogrammetry

An SEM quantitative stereophotogrammetry technique (SQS), was developed to describe composite fracture surfaces. This technique consists of a three-dimensional plot of the fracture surface created by digitally analysing SEM stereographic pairs (companion photographs taken of the same image at different angles of incidence to the object). From this three-dimensional surface roughness plot, a surface roughness parameter may then be calculated. This technique is a non-destructive method, except for any beam damage produced on the fracture surface by prolonged electron beam exposure.

A CamScan Series 4 SEM with a Kevex Super 8000 image analyser and corresponding Kevex Advanced Imaging software was employed to obtain the surface roughness data. Stereographic pairs were taken at tilt angles of $\pm 4°$ from normal incidence. The plane of the photos was parallel to the crack propagation direction. Each of the two SEM images of the stereo pair was then processed using the Kevex image analyser, where they were digitized by brightness level into 512×256 pixel arrays. Fig. 2 shows a scanning electron micrograph and its associated digital image, for a composite containing 40 vol % SiC whiskers hot pressed at 1600 °C.

Once digital images had been generated, they were then analysed using the Stereo Spot program which is part of the Kevex Advanced Imaging software package. This program uses differences in the coordinates of a feature present in each image and the overall tilt angle between the two stereo images to calculate a vertical height measurement normal to the plane of the photo. This height measurement is made relative to a zero point set by the user. The area of the specimen employed to produce each image was $378\ \mu m \times 312\ \mu m$ at a magnification $\times 250$.

The height measurements in micrometres and the x and y coordinates in pixels, which are subsequently converted into micrometres, were used to create a three-dimensional surface roughness map, using the Displaa graphics software package. A random array of x, y, and z values were used by the software to calculate a regular matrix of x, y, and z values using linear interpolation. Two to three hundred data points were employed to create each plot.

2.3. Surface roughness parameter

The fundamental approach of quantitative surface roughness parameters is shown in Fig. 3, from Banerji [4]. For any irregular surface, both a lineal roughness parameter, R_L, and a surface roughness parameter, R_S, may be defined as follows

$$R_L = L_t/L' \quad (1)$$

$$R_S = S_t/A' \quad (2)$$

where L_t is the true profile length, L' the projected profile length, S_t the true surface area, and A' the projected surface area. The surface roughness parameter is related to the lineal roughness parameter as [4]

$$R_S = (4/\pi)(R_L - 1) + 1 \quad (3)$$

For a flat surface $R_S = 1$, whereas $R_S > 1$ for a rough surface.

A computer program was developed which employed x–y–z surface roughness map array data to calcu-

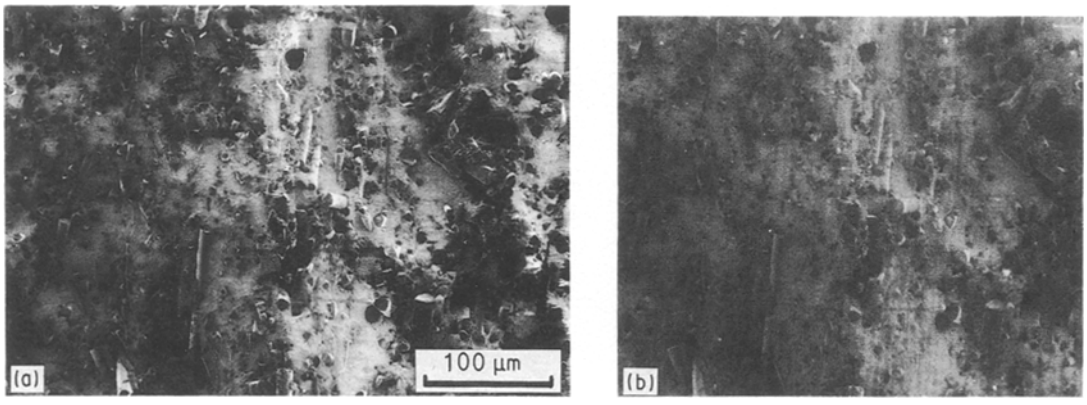

Figure 2 (a) Scanning electron micrograph and (b) its associated digital image, for a composite containing 40 vol % SiC whiskers hot pressed at 1600 °C.

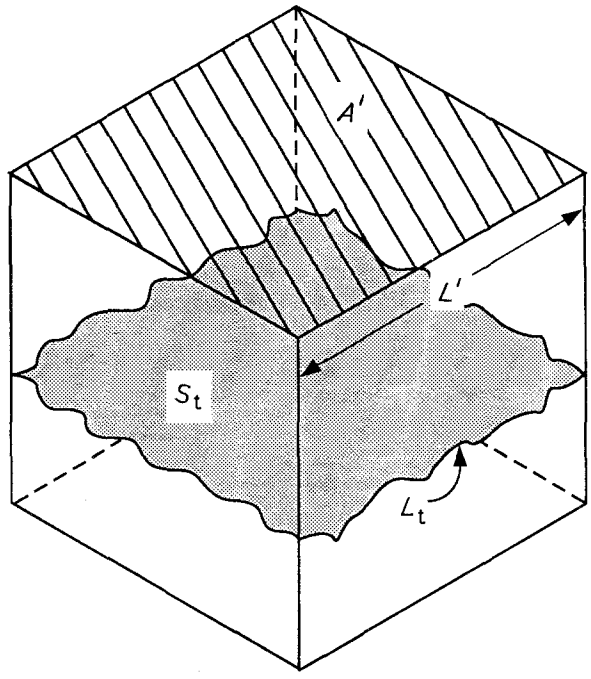

Figure 3 Fundamental approach of quantitative surface roughness parameters. From [4].

late the surface roughness parameter, R_S. This was done by finding the distance between each point along a grid line in the surface roughness map. The sum of these inter-point distances is then equal to the true length L_t. The true length for each grid line was then converted into the linear roughness parameter, R_L, by dividing by the projected length (i.e. the length of the pixel array in micrometres). Then, Equation 3 was employed to obtain the surface roughness parameter, R_S.

The surface roughness parameter obtained in this manner does not correspond one-to-one to the surface roughness parameter of the actual fracture surface. The difference arises due to the necessary minor changes made in the data to create the regular array used to plot the surface roughness map. However, the calculated surface roughness parameter is considered to be a good approximation to the true value.

2.4. Profilometer surface finish

In order to obtain an additional measurement of fracture surface roughness for comparison to the SQS technique, composite fracture surfaces were also measured using a mechanical profilometer surface finish technique. In this method, a fine-tipped stylus under a light mechanical load is slowly translated across the fracture surface, and vertical displacements are measured during this translation.

The method yields a root-mean-squared (RMS) surface finish value given in micrometres as an index of surface roughness. All profilometer measurements were made on the same fracture surface specimens used for the SQS measurements, in a direction parallel to the hot-pressing direction. Measurements were also made on pure Si_3N_4 matrix specimens hot pressed at 1600 and 1750 °C.

2.5. SEM of matching fracture surfaces

A limited investigation of matching fracture surfaces was conducted in order to provide additional information concerning the extent of whisker pull-out occurring in the VLS SiC whisker–Si_3N_4 matrix composites, and its potential contribution to fracture surface roughness. The matching fracture surfaces of chevron-notched bend specimens of selected composite specimens were examined, and micrographs of the same fracture area taken at the same magnification. This allowed for a straight forward evaluation of the extent of whisker pull-out.

3. Results

SQS surface roughness maps for the VLS SiC whisker–Si_3N_4 matrix composites are shown in Fig. 4. Table II compares surface roughness parameters obtained through Equations 1–3 with profilometer surface finish values, and measured fracture toughness values for the composites. Fig. 5 plots SQS surface roughness parameters versus associated profilometer surface finish values. Figs 6 and 7 show composite fracture toughness as a function of profilometer surface finish and SQS surface roughness parameter, respectively. Fig. 8 shows matching fracture surfaces for a composite containing 10 vol % SiC whiskers, which was hot pressed at 1750 °C.

It is evident from the fracture surface contours in Fig. 4 that, at constant 10 vol % whiskers, the fracture surface roughness tends to increase with increasing hot-pressing temperature between 1600 and 1750 °C. This is confirmed by both SQS surface roughness parameter and profilometer surface finish values in Table II. However, no significant increase in surface roughness is detected between 1750 and 1850 °C. For a constant hot-pressing temperature of 1600 °C, the surface roughness increases markedly between 10% and 40% volume fraction SiC whiskers.

In order to determine if there was a distinct functional relationship between SQS surface roughness parameter and profilometer surface finish, the data were plotted against each other in Fig. 5. As may be seen, there is the suggestion of a linear relationship for composites hot pressed at 1750 and 1850 °C. However, the relationship for 1600 °C data is not linear.

The plots of fracture toughness versus profilometer surface finish (Fig. 6) and SQS surface roughness parameter (Fig. 7) show that fracture toughness increases with increasing fracture surface roughness. An approximate linear relationship is suggested, except for the VLS SiC whisker–Si_3N_4 matrix composite hot pressed at 1850 °C which exhibited a high fracture toughness value of 12.6 MPa $m^{1/2}$. The slopes for composites hot pressed at 1600 and 1750 °C are roughly similar.

The matching fracture surfaces in Fig. 8 show that little whisker pull-out occurred for 1750 °C hot pressing. Similar observations were noted for 1600 and

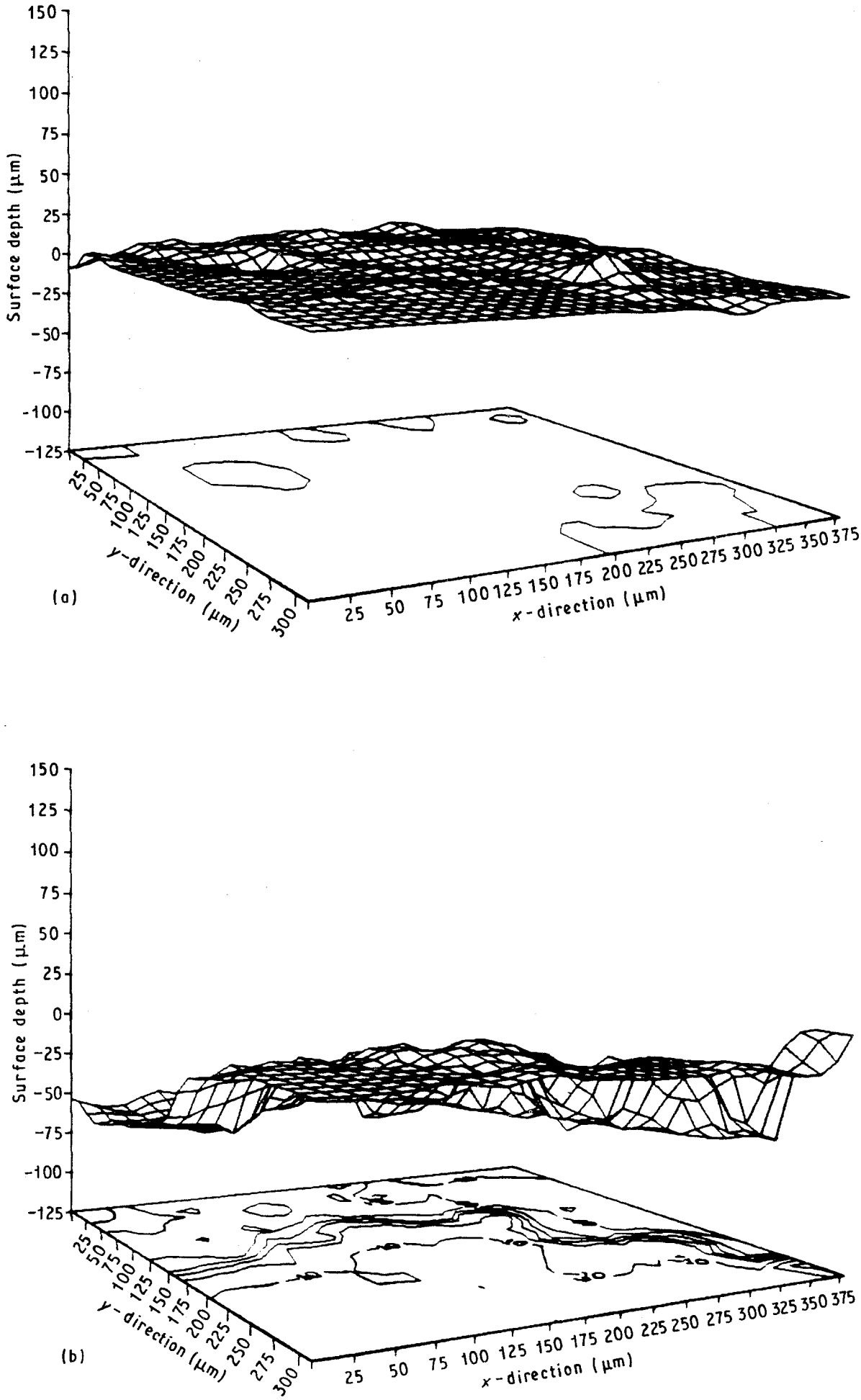

Figure 4 SQS surface roughness maps for VLS SiC whisker–Si_3N_4 composites. 10 vol % SiC composite, hot pressed at (a) 1600 °C, (b) 1750 °C, (c) 1850 °C, (d) 40 vol % SiC composite hot pressed at 1600 °C.

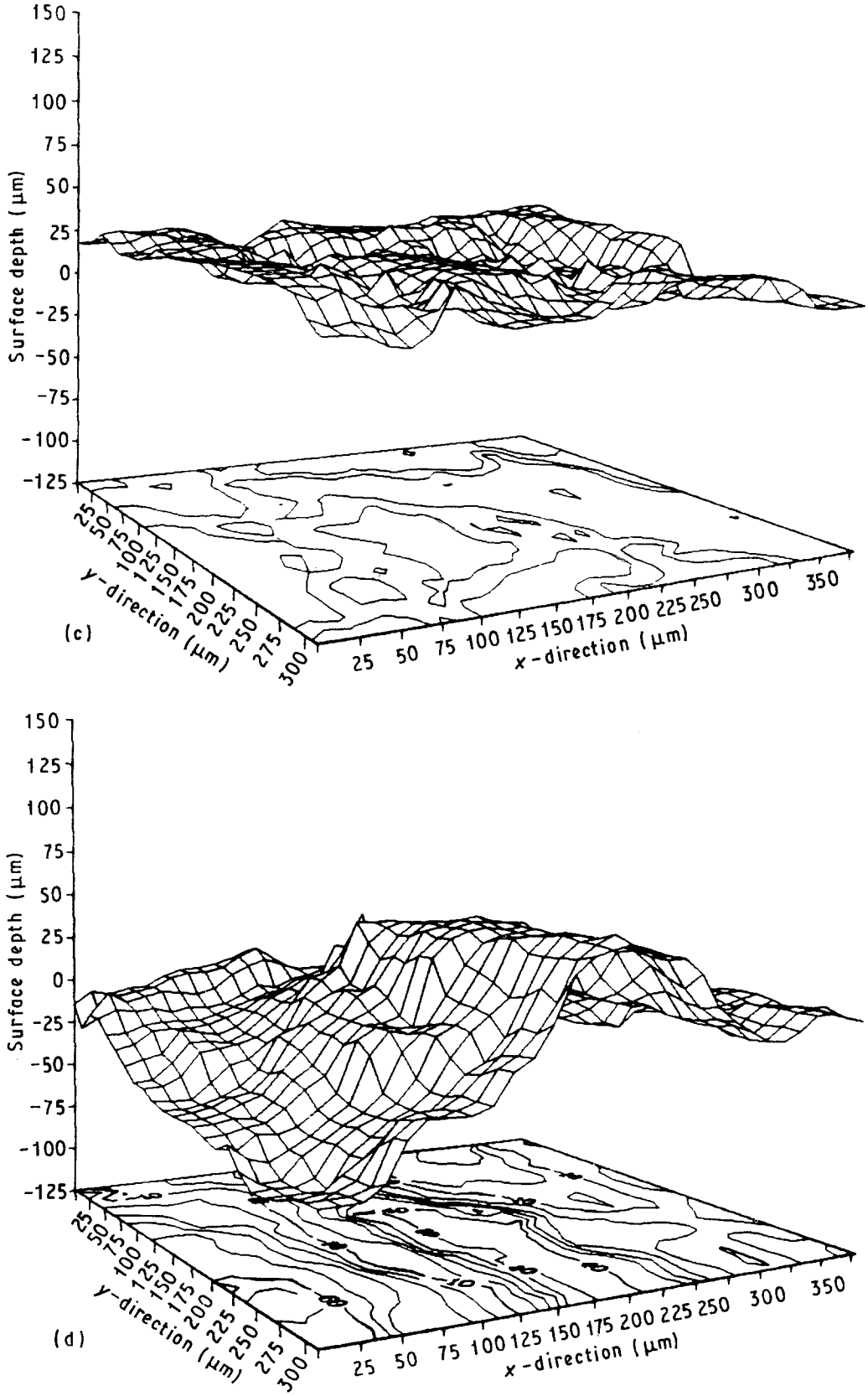

Figure 4 (Continued).

1850 °C hot pressing. As may be seen in Fig. 8, whiskers oriented parallel to the fracture surface exhibited decohesion of the whisker–matrix interfacial bond.

4. Discussion

The increase in surface roughness observed between composites hot pressed at 1600 °C and those hot pressed at 1750 and 1850 °C may be associated with the following aspects. The first aspect is that the Si_3N_4 matrix crystal structure was primarily alpha at 1600 °C, but changed to primarily beta at 1750 and 1850 °C. This transformation results in a matrix grain shape change from equiaxed to elongated, which has been reported to increase the fracture toughness of pure Si_3N_4 [5]. Such a change in grain morphology

TABLE II Comparison of surface roughness parameters and surface finish values to fracture toughness

Hot-pressing temperature (°C)	SiC whiskers (vol %)	Surface roughness parameter	Surface finish (RMS μm)	Fracture toughness (MPa $m^{1/2}$)
1600	0	1.000	0.240	5.7
1750	0	1.000	0.361	7.6
1600	10	1.163	0.278	6.1
1750	10	1.264	0.655	9.8
1850	10	1.237	0.622	12.6
1600	40	1.383	0.967	9.4

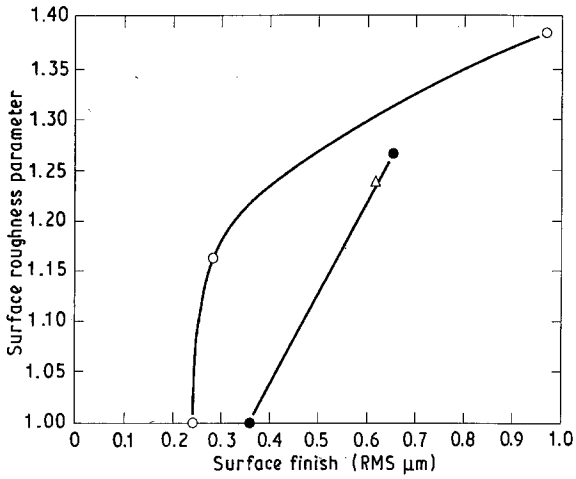

Figure 5 Plot of SQS surface roughness parameters versus associated profilometer surface finish values; hot pressed at (○) 1600 °C, (●) 1750 °C, (△) 1850 °C.

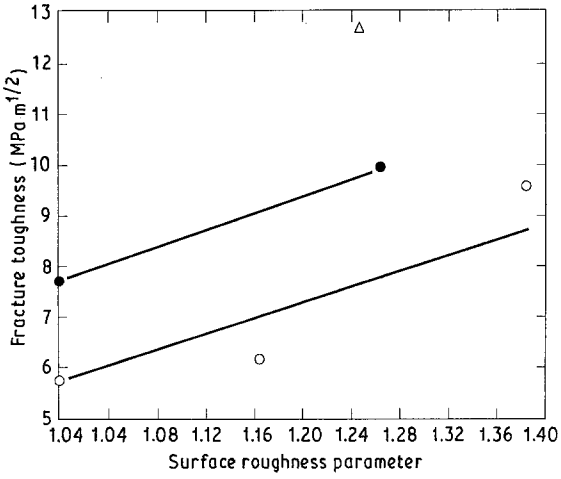

Figure 7 Composite fracture toughness as a function of SQS surface roughness parameter; hot pressed at (○) 1600 °C, (●) 1750 °C, (△) 1850 °C.

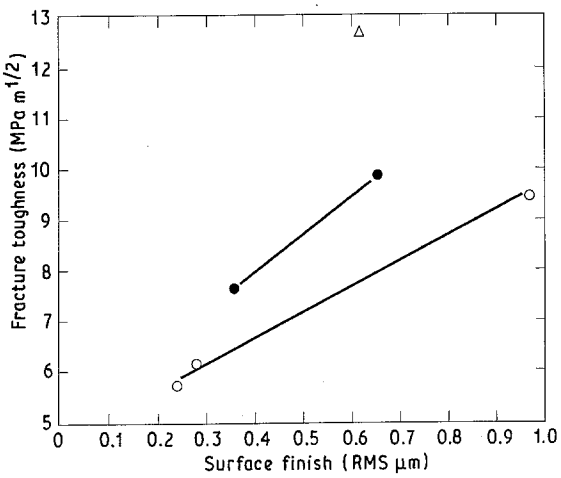

Figure 6 Composite fracture toughness as a function of profilometer surface finish; hot pressed at (○) 1600 °C, (●) 1750 °C, (△) 1850 °C.

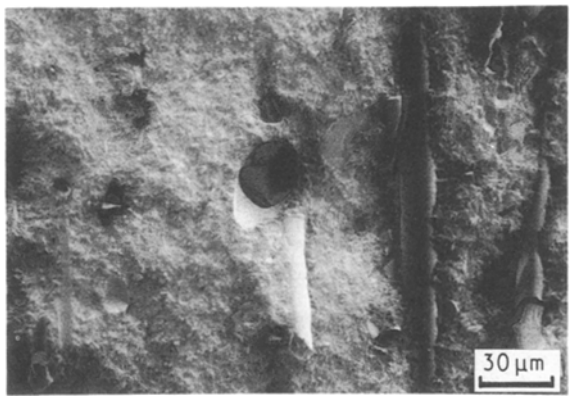

Figure 8 Matching fracture surfaces of composite containing 10 vol % SiC whiskers hot pressed at 1750 °C.

should increase surface roughness, for intergranular fracture modes. Secondly, the properties of the glassy grain-boundary phase present in the composites due to the MgO densification aid may be a function of the hot-pressing temperature. This grain-boundary glassy phase has been shown to crystallize upon cooling from the hot-pressing temperatures [6]. The propensity for crystallization has been found to increase as the hot-pressing temperature is increased. This effect may be explained by changes in the amount of glass and its

composition with the hot-pressing temperature. Such changes in composition or amount of glass present will change the likelihood of crystallization during cooling.

Such crystallization would tend to lower the whisker–matrix interfacial bond strength [7, 8]. The lower interfacial strength may be explained as follows. The high atomic mobility of atoms present in the glassy grain boundary phase might allow the glass atoms to be in close registry with the atoms of the whisker or matrix. This would lead to a strong interfacial bond between the whiskers, glassy phase, and matrix. When the glass crystallizes, the arrangement of atoms in the crystallized glass is governed by the crystal structure of the glass, and these atoms are not as free to move around to accommodate the crystal structures of the whiskers and matrix. This would lead to a lower interfacial strength as compared to the glassy interface, because of the increased lack of atomic registry at the interface. The strength of the interface may also be affected by stresses that develop during crystallization and cooling. A decrease in the interfacial strength would tend to increase the extent of crack deflection [9, 10]. The interfacial strength must not be decreased by a large amount because extensive whisker pull-out was not observed. If the interfacial strength was greatly reduced by the crystallization of the glassy grain-boundary phase, the whiskers would readily debond and pull out. This was not observed in the composites studied in this investigation.

Nutt and Phillips [6] have performed TEM studies of VLS SiC whisker–Si_3N_4 matrix composites hot pressed at 1600 and 1850 °C. They noted the presence of an Mg–Sialon interfacial phase at some whisker–matrix interfaces, for both hot-pressing temperatures. However, the overall extent of crystallization at the two hot-pressing temperatures was not quantitatively assessed. The VLS SiC whiskers showed no signs of reaction with the Si_3N_4 matrix for composites hot pressed at 1600 °C. However, for 1850 °C composites, a slight interfacial reaction was observed. This reaction took the form of approximately 10 nm notches at the interface on the whisker side, presumably due to some dissolution of the SiC at the 1850 °C hot-pressing temperature. This reaction between the SiC whiskers and the Si_3N_4 matrix supports the argument that the interface glass composition changes with hot-pressing temperature. These TEM observations suggest that the SiC whisker–Si_3N_4 matrix interface will become weaker with increasing hot-pressing temperature.

Recently, Hutchinson and co-worker [9, 10] have evaluated the fracture mechanics conditions necessary for crack deflection at an interface. The criterion deduced for crack deflection at an interface is that the toughness of the interface should be one-quarter or less the toughness of the reinforcement. This makes it clear that the extent of crack deflection will increase as the interfacial bond strength becomes weaker. Thus, the fracture toughness should increase with decreasing interfacial strength.

For 1600 °C hot pressing, both the SQS surface roughness and profilometer surface finish values increased significantly between 10 and 40 vol % SiC whiskers. It is likely that some of this increase is attributable to increasing amounts of crack deflection with increasing whisker volume fraction. However, another contributing factor is the fact that the composite hot pressed with 40 vol % whiskers was only 91% dense, while the 10 vol % whisker composite was 99.5% dense. Thus, it is likely that residual porosity also made a contribution to the measured surface roughness.

In Figs 6 and 7, the composite hot pressed at 1850 °C, which exhibited a measured fracture toughness of 12.6 MPa $m^{1/2}$, falls significantly above the trend curves of the other composites for fracture toughness versus surface roughness. Thus, the higher toughness value measured was not associated with an accompanying higher value of surface roughness. It is interesting to note that a second specimen hot pressed at 1850 °C showed a fracture toughness value of 10 MPa $m^{1/2}$. If this toughness value were employed at the same level of surface roughness, the 1850 °C data point would fall close to the 1750 °C trend curves in Figs 6 and 7. These observations indicate that the higher absolute toughness level observed for the one 1850 °C specimen must be related to factors other than surface roughness.

In general terms, the data in Figs 6 and 7 establish quantitatively that increased surface roughness accompanies the increased fracture toughness of VLS SiC whisker–Si_3N_4 matrix composites. Because significant contributions from whisker pull-out do not occur (Fig. 8), the toughening mechanisms operative must be either crack deflection [2], crack bridging [3], or a combination of these two mechanisms. The crack deflection mechanism clearly incorporates increased surface roughness as an intimate aspect of the toughening mechanism, because the crack must circumvent the reinforcement obstacles by tilting and/or twisting. As presently envisioned, the crack bridging mechanism leads to toughening due to the fact that the bridges limit the crack opening displacement and hence the stress intensity factor at the crack tip. Increased surface roughness is not necessarily associated with the crack bridging mechanism. Thus, the present results may point more strongly towards crack deflection than crack bridging as the dominant toughening mechanism, although the simultaneous operation of crack bridging is also likely.

5. Conclusions

An SEM quantitative stereophotogrammetry (SQS) technique was developed and applied to analyse the fracture surfaces of VLS SiC whisker–Si_3N_4 matrix composite fracture toughness specimens. Application of the SQS technique in conjunction with mechanical surface profilometer measurements has quantitatively established that increased surface roughness is associated with increased fracture toughness for these composites. Si_3N_4 grain morphology and whisker/matrix interfacial bond strength appear to contribute to composite surface roughness.

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