JOURNAL OF MATERIALS SCIENCE 18 (1983) 3099–3104

Mechanical behaviour of TiC film coated on molybdenum by magnetron sputtering

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The TiC film, which is coated on molybdenum by magnetron-sputtering, is analysed after the molybdenum substrate is tensile-tested to rupture at 300 to 1070 K. At 300 K some portion of the film exfoliated during the molybdenum substrate deformation. The degree of exfoliation is proportional to the substrate strain up to about 30% elongation, and is proportional to the square root of the film thickness. The maximum shear stress which is generated at the interface between the film and the substrate during the deformation is estimated by the measurement of the distance between cracks. From the estimated maximum shear stress, the adhesive strength of the present TiC film is evaluated to be about 400 MN m^{-2}.

1. Introduction

Titanium carbide (TiC)-coated molybdenum is one of the strongest candidates for the first-wall components in the Tokamak-type nuclear fusion reactor in Japan. The major apprehension about the use of this composite is that the surface TiC film may exfoliate during the plasma discharge operation [1]. When the TiC film exfoliates, the high-Z (Z is the atomic number) molybdenum will face the hot fusion plasma. The sputtered molybdenum atoms will then contaminate and cool the plasma with the result that the critical condition for the nuclear fusion reaction cannot be attained [2].

The main cause of the exfoliation of the coating film will be the thermal stress generated by the transient and abrupt temperature increase at the first-wall surface [1]. To avoid such disastrous damage to the coating film, one must coat molybdenum with a strongly adhesive film. Also it is necessary to study the behaviour of the coating film under stress.

Here, we report the behaviour of the TiC film deposited on molybdenum by magnetron-sputtering when the substrate is deformed. We also try to evaluate the adhesive strength of the coating TiC film.

2. Experimental procedures

Detailed procedures of the specimen preparation and of the tensile test can be found in the previous report [3]. The detailed analysis of the characteristics of the coating TiC film can be found elsewhere [4].

The TiC film, whose chemical composition is $Ti_{0.4}C_{0.6}$, having the single phase of TiC without the graphite phase [4], and having the (111) preferred orientation normal to the substrate surface, were deposited on fully annealed molybdenum substrate at 870 K by magnetron-sputtering. The thickness of the coating TiC film was 1.2 to 6 μm. The tensile test was conducted at 300, 670, 870 and 1070 K at strain rates from 1.4×10^{-3} to 4.2×10^{-2} sec^{-1}.

The major results were obtained for 6 μm thick TiC-coated specimens at strain rates of 1.4×10^{-3} sec^{-1}. In the following, the TiC film thickness and the strain rates are the above mentioned values, unless they are particularly quoted.

After the composite was tensile-tested to rupture, the surface TiC film was examined by scanning electron microscopy (SEM). Many cracks were found, mainly normal to the tensile direction in the TiC film. The distance between two cracks

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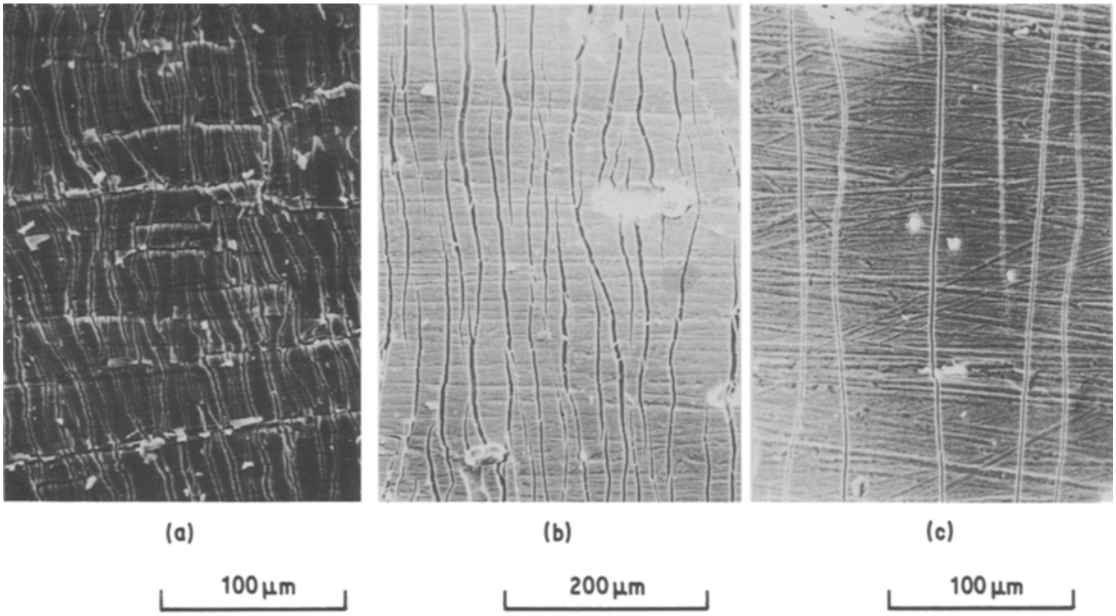

Figure 1 Scanning electron micrographs of the surface of a TiC film after molybdenum substrate is ruptured at 300 K. (a) Thickness of film = 1.2 μm, strain rate = 1.4 × 10^{-3} sec^{-1}; (b) thickness of film = 6 μm, strain rate = 1.4 × 10^{-3} sec^{-1}; and (c) thickness of film = 6 μm, strain rate = 4.2 × 10^{-2} sec^{-1}.

(the interdistance) was measured on the SEM photographs. About 20 to 30 interdistances were measured and averaged. In the following, the interdistance of cracks refers to this averaged value.

The amount of exfoliation of the TiC film during the tensile test was evaluated by the weight change. The specimen was weighed before and after the tensile test by an electromicrobalance (Mettler ME22). The weight loss during the tensile test was divided by the total weight of the TiC film to obtain the degree of exfoliation.

Some of the composites were heat treated at 1500 K for 5 h after coating. This heat treatment caused molybdenum diffusion into the TiC film and Mo_2C formed at the interface between the molybdenum substrate and the TiC film [4]. The formation of this diffusion zone would improve the adhesive strength of the film to the substrate.

3. Results

The surface morphology of the TiC film after the tensile test is shown in Figs. 1, 2 and 3. The rupture elongation of the molybdenum substrate is about 30% in all cases. One can see many cracks normal to the tensile direction. The interdistance of cracks is found to be specific to each specimen. The interdistance of cracks is plotted as a function of distance from the ruptured surface in Fig. 4. One can see that the interdistance remains nearly constant in the gauge region. Outside the gauge region (where the molybdenum substrates deform far less than inside the gauge region), the interdistance becomes larger than that inside the gauge region.

The thicker TiC film has longer interdistances, as one can see in Fig. 4 and by comparing Figs. 1a and b. Figs. 1a and b show the surface morphologies of 1.2 and 6 m thick TiC films tensile-tested at 300 K at a strain rate of 1.4 x 10^{-3} sec^{-1}. Fig. 5 shows the interdistance of cracks as a function of the film thickness at 300 K. The interdistance is found to be proportional to the film thickness.

Fig. 6 shows the temperature dependence of the interdistance of cracks. Figs. 1b and 2a to c depict the temperature dependence of the morphology of cracks. With the increase in temperature, the interdistance of cracks increases and the cracks become straight.

The heat-treated specimen has a different configuration of cracks from that in the as-deposited ones. In Figs. 3a and b, the cracks in the heat-treated film are found to be irregular with some longitudinal cracks.

The interdistance of cracks in the heat-treated film is also plotted in Fig. 6, as a closed circle, at 300 and 1070 K. The heat treatment, namely the improvement of the adhesion of the film does not seem to change the interdistance of cracks.

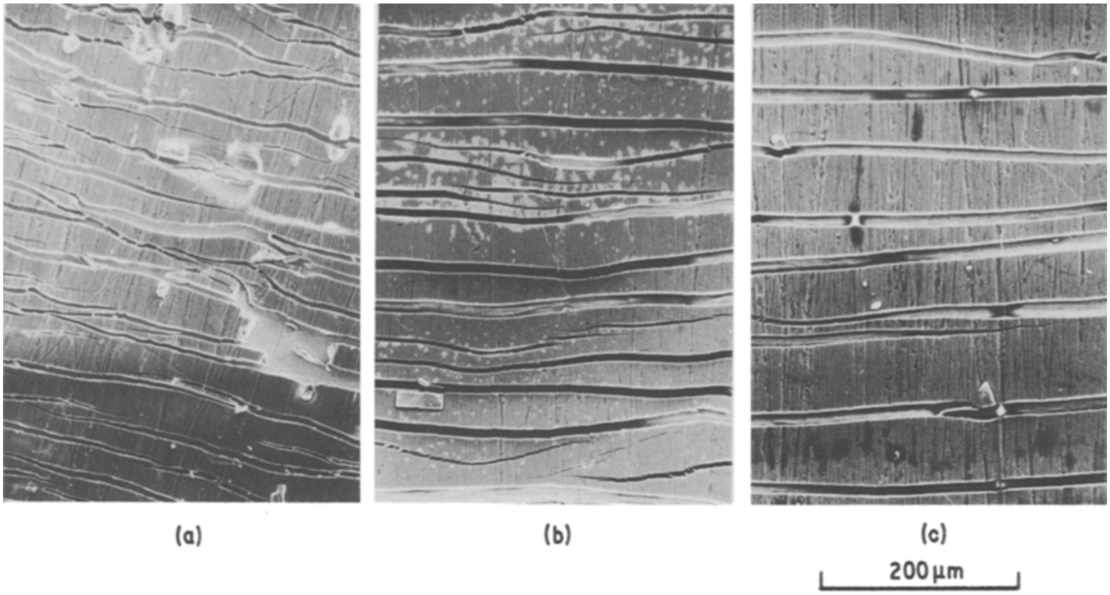

Figure 2 Scanning electron micrographs of the surface of a TiC film (thickness = 6 μm) after molybdenum substrate is ruptured at elevated temperatures at a strain rate of 1.4×10^{-3} sec^{-1}: (a) 670 K, (b) 870 K, and (c) 1070 K.

The degree of exfoliation of the film during deformation can be seen in Fig. 7, where the strain rates are different in the different data points. Neglecting the strain rate effect, the degree of exfoliation is found to be proportional to the plastic strain of the molybdenum substrate. The closed circles in Fig. 7 represent results at higher temperatures. The higher temperature tensile tests caused a lesser degree of exfoliation.

The degree of exfoliation is also plotted as a function of the thickness of the TiC film in Fig. 8. The degree of exfoliation depends strongly on the film thickness. In the present experiment, the degree of exfoliation seems to be proportional to the square root of the film thickness.

4. Discussion

As discussed in the previous paper [3], the present

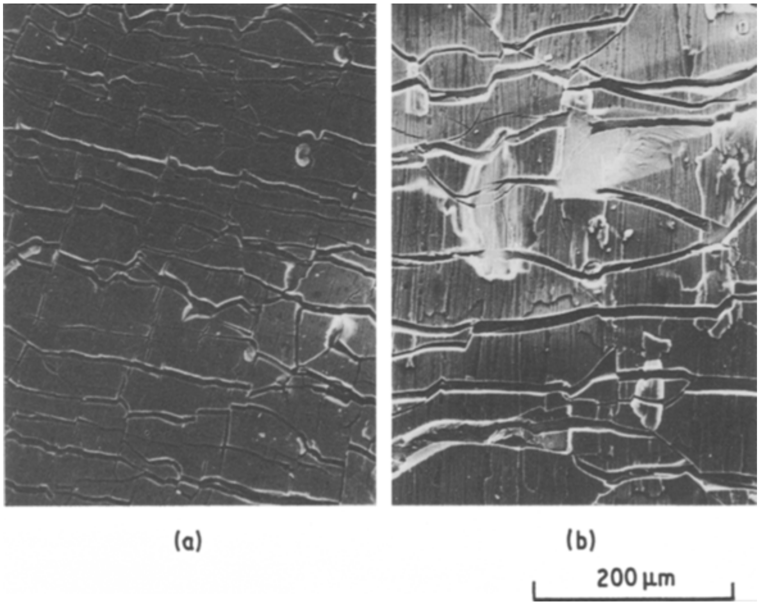

Figure 3 Scanning electron micrographs of the surface of a TiC film, which is heat treated at 1500 K for 5 h before the tensile test. Film thickness = 6 μm, strain rate = 1.4×10^{-3} sec^{-1}: (a) 300 K, and (b) 1070 K.

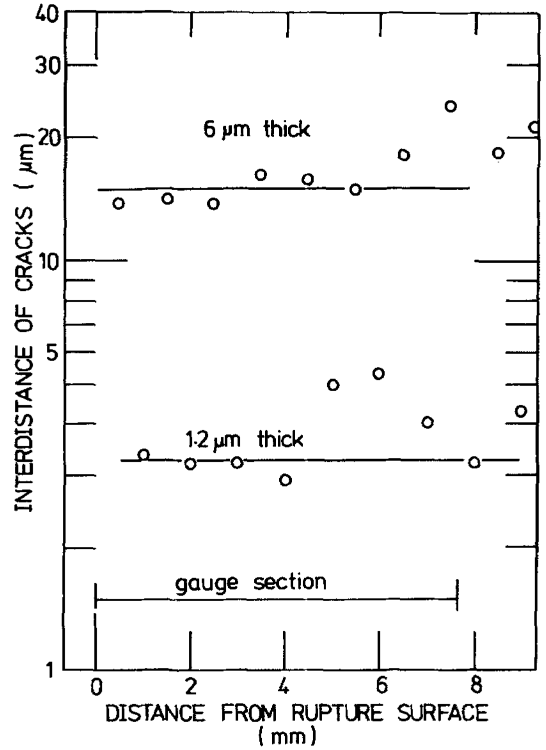

Figure 4 Interdistance of cracks as a function of the distance from the ruptured surface on tensile-tested TiC film at 300 K and at a strain rate of 1.4×10^{-3} sec^{-1}.

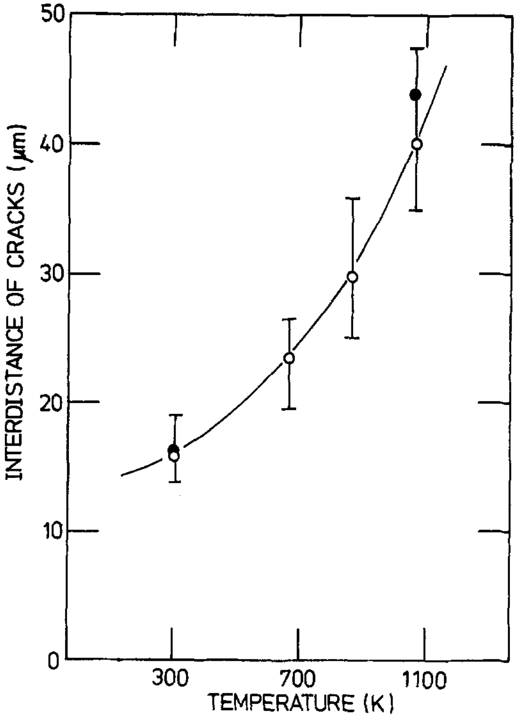

Figure 6 Interdistance of cracks as a function of tensile-test temperature at a strain rate of 1.4×10^{-3} sec^{-1}: open circle – as-deposited specimen; closed circle – heat-treated specimen after deposition.

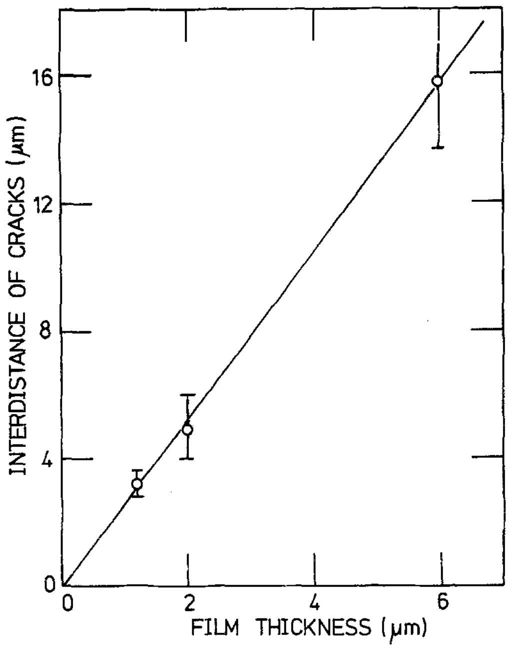

Figure 5 Interdistance of cracks as a function of film thickness at 300 K and at a strain rate of 1.4×10^{-3} sec^{-1}.

TiC film is adhesive to the molybdenum substrate. In most cases, the exfoliation of the film is negligible during the tensile test in which the molybdenum substrate deforms more than 30%. By the constrained effect of the TiC film, the apparent strength of the molybdenum substrate increases [3].

When the stress is applied to the molybdenum substrate, the shear stress is generated along the interface between the film and substrate. The shear stress, τ, will be carried by the bulk stress, σ, through the following relation,

$$La\tau = ta\sigma \qquad (1)$$

Here, L, a and t are interdistances of two cracks, width of the film and the film thickness, respectively.

When

$$La\tau > ta\sigma \qquad (2)$$

the new crack is formed between the previous two cracks and finally the maximum shear stress, τ_m, and the strength of the film, σ_t, will equilibrate each other in the following relation, assuming that the new crack is always formed just at the

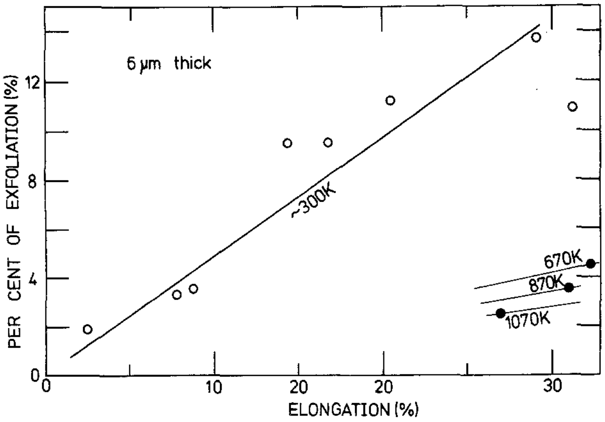

Figure 7 Degree of exfoliation as a function of substrate elongation: open circle – at 300 K; closed circle – at elevated temperatures.

middle of the previous two cracks:

$$2L\tau = t\sigma_t \qquad (3)$$

Actually the strength of the film, σ_t, is the fracture strength of the TiC film, σ_{tf}.

In Relation 3 the interdistance of cracks should be proportional to the thickness of the film, t, when τ_m and σ_{tf} are constant. This is the case in the present result, shown in Fig. 5.

In the previous paper [3], we estimated the fracture strength, σ_{tf}, of the present TiC film. Using those values and Relation 3, we estimated the maximum shear stress, τ_m. The results are shown in Fig. 9.

When the maximum shear stress exceeds the adhesive strength of the film, τ_a, the film will exfoliate. In the present experiment, the exfoliation can be observed appreciably only at 300 K. From this result the adhesive strength of the present film is estimated to be about 400 MN m^{-2} (the maximum shear stress at 300 K).

At elevated temperatures, the maximum shear stress along the interface decreases rapidly, as shown in Fig. 9. This decrease of the maximum shear stress will be the reason why the degree of the film exfoliation decreases at the higher temperatures shown in Fig. 7. Here, we assumed that the adhesive strength of the film would not have a strong temperature dependence.

In the present experiment, the adhesive strength of the film is considered to exceed the maximum shear stress along the interface. So, further improvement of the adhesive strength by heat treatment did not affect the results seriously.

Also, the heat treatment did not affect the

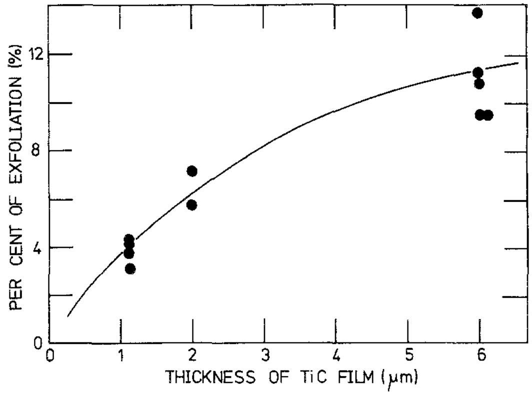

Figure 8 Degree of exfoliation as a function of film thickness at 300 K and at a strain rate of 1.4 × 10^{-3} sec^{-1}.

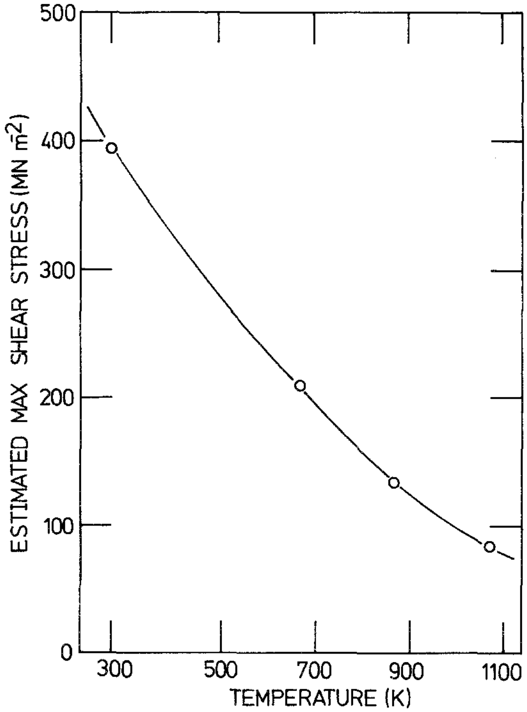

Figure 9 Estimated maximum shear stress along the interface as a function of temperature.

mechanical properties of the TiC-coated molybdenum. The amount of the increase in the 0.2% proof strength is about the same in the as-deposited and non-heat-treated composite as reported in the previous report [3]. This result supports the assumption that the adhesive stress of the film exceeds the maximum shear stress and that the strengthening effect of the TiC coating is governed by the bulk strength of the film.

At lower temperatures, where the shear stress may exceed the adhesive strength, the effect of the heat treatment may be apparent.

High-temperature tensile test and tensile tests at higher strain rates results in straight crack formation, as one can see in Fig. 1c and Figs. 2a to c. In those cases, cracks have a wider interdistance than in the films tensile-tested at 300 K at a strain rate of 1.4×10^{-3} $\sec^{-1}$.

So, we can summarize the results concerning the crack morphology, that the crack morphology becomes straight with the increase in interdistance of cracks; in other words, with the increase of σ/τ ($= 2L/t$). The fracture mode of the TiC film may depend on σ/τ, but further investigation is needed to make this clear.

5. Conclusions

The TiC film coated on molybdenum was analysed after the molybdenum substrate was tensile-tested to rupture at 300 to 1070 K. The TiC film was deposited on molybdenum by magnetron-sputtering. At 300 K, some portion of the TiC film exfoliated during the deformation of the molybdenum substrate. The degree of exfoliation is proportional to the substrate strain up to about 30% elongation, and is proportional to the square root of the film thickness.

When the molybdenum substrate was deformed to rupture, many cracks were formed in the TiC film, mainly normal to the tensile direction. From the measurement of the interdistance of two cracks we estimated the maximum shear stress generated at the interface between the TiC film and the molybdenum substrate.

The estimated shear stress decreases with the increase in deformation temperature. The degree of exfoliation decreases rapidly with the increase of the deformation temperature. The improvement of the adhesive strength by heat treatment did not affect the results seriously.

From these results the adhesive strength of the present TiC film is estimated to be about 400 MN m^{-2} (the estimated maximum shear stress at 300 K). The crack configuration changes from wavy to straight with the increase in the interdistance of cracks.

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Received 6 January
and accepted 24 February 1983