Available online at www.sciencedirect.com
ScienceDirect
J. Mater. Sci. Technol., 2010, **26**(1), 65-68.
JMST
www.JMST.org

Mechanical Properties of the $TaSi_2$ Fibers by Nanoindentation

Chunjuan Cui[1,2)†], *Jun Zhang*[1)], *Lin Liu*[1)] *and Hengzhi Fu*[1)]
1) State Key Laboratory of Solidification Processing, Northwestern Polytechnical University, Xi′an 710072, China
2) School of Metallurgical Engineering, Xi′an University of Architecture and Technology, Xi′an 710055, China
[Manuscript received November 7, 2008, in revised form May 18, 2009]

The Si-$TaSi_2$ eutectic *in situ* composite, which has highly-aligned and uniformly-distributed $TaSi_2$ fibers in the Si matrix, can be obtained when the solidification rate changes from 0.3 to 9.0 mm/min. It is very interesting that one or two $TaSi_2$ fibers are curved when the solidification rate reaches 6.0 mm/min, although it is very brittle in general. The formation mechanism of the curved fiber is discussed and mechanical properties of the $TaSi_2$ fibers are examined by nanoindentation. It is found that the hardness and the elastic modulus of the bended $TaSi_2$ fiber are much higher than that of the straight $TaSi_2$ fiber. Moreover, the reasons why the mechanical properties of the straight $TaSi_2$ fiber are different from that of the curved $TaSi_2$ fiber are discussed. This can be ascribed to internal stress which results from mismatch of the thermal expansion coefficients of the two phases and different crystallographic orientations.

KEY WORDS: Si-$TaSi_2$ eutectic *in situ* composite; Directional solidification; Mechanical properties; Nanoindentation

1. Introduction

$TaSi_2$ is one of the attractive candidates for microelectronic industry due to good electron conductivity, relatively low work function and good combination with Si[1–3]. Si-$TaSi_2$ eutectic *in situ* composite is one kind of promising field emission materials. Ditchek *et al.*[4,5] have successfully prepared the Si-$TaSi_2$ eutectic *in situ* composite by Czochralski technique. The Si-$TaSi_2$ eutectic *in situ* composites with better field emission properties have been obtained by electron beam floating melting (EBFZM) technique on the basis of Czochralski method at a solidification rate R=0.3∼9.0 mm/min[6]. Typical microstructure of the Si-$TaSi_2$ is shown in Fig. 1(a). It can be seen that the $TaSi_2$ fibers are straight commonly. It is very interesting to observe that one or two $TaSi_2$ fibers are curved when the solidification rate reaches 6.0 mm/min as shown in Fig. 1(b). As is known, $TaSi_2$ has the C40 structure, low fracture toughness and ductility at low temperature, and less elastic anisotropy[1]. Therefore it is very brittle in general. This phenomenon arouses our interest. In the present experiment, the mechanical properties of the $TaSi_2$ fibers are studied by nanoindentation. Novel nano-indentation technology, which can record the continuous load-depth history in micro/nano scale precisely, has been widely used for investigating mechanical properties of carbon fibers, films, coatings *etc.*[7–9]. The present investigation can help us know this material well and provide some guidance on the fabrication of the Si-$TaSi_2$ field emitter arrays synchronously.

2. Experimental

The longitudinal section of the Si-$TaSi_2$ eutectic prepared at the solidification rate R=6.0 mm/min is mechanically polished, and the average surface rough-

† Corresponding author. Prof., Ph.D.; Tel.: +86 29 82205104; Fax: +86 29 82202931; E-mail address: cjcui_16@yahoo.com (C.J. Cui).

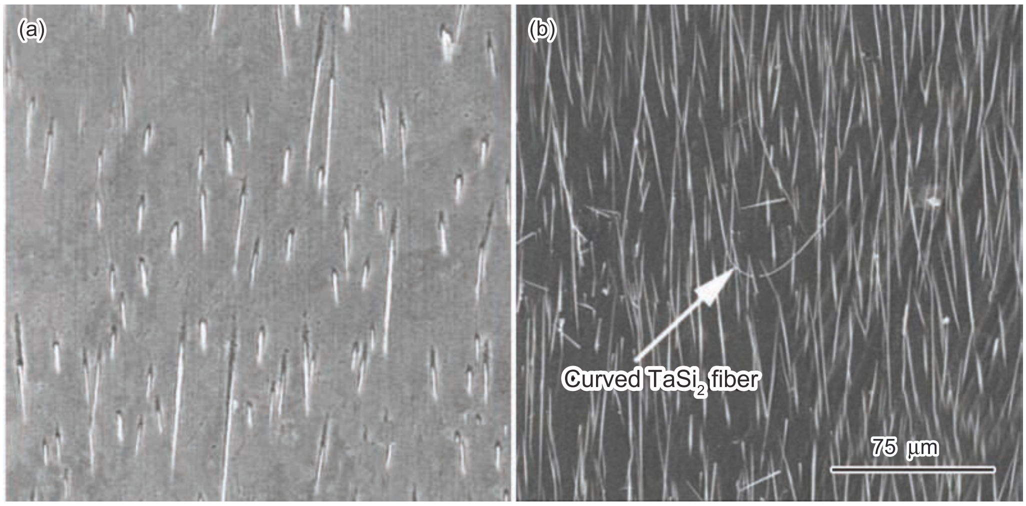

Fig. 1 Microstructure of the $TaSi_2$ fibers embedded in the Si matrix: (a) typical straight fibers; (b) curved $TaSi_2$ fiber at the solidification rate R=6.0 mm/min

ness of each polished specimen was less than 100 nm. Then the sample is etched with a HF/HNO_3 corrosive to expose the $TaSi_2$ fibers. The tiny zone in which the bended $TaSi_2$ fiber exists is located by HITACHI S-570 scanning electronic microscopy (SEM) and cut by diamond chipper. The indentation tests are made by using a Nano Indenter DCM with a Berkovich triangular diamond indenter at a loading rate of 0.05 mN/s. The location precision of the nanoindentaion is 2 nm, the diameter of the $TaSi_2$ fiber is in the neighbourhood of 0.74∼0.82 μm[10]. Therefore, the results are credible.

3. Results and Discussion

3.1 Formation mechanism of the curved fibers

A possible explanation for the formation of the curved fiber is the difference between thermal expansion coefficients of the Si and $TaSi_2$ phases (Si: 2.6×10^{-6} K^{-1}[11,12], $TaSi_2$: 14.0×10^{-6} K^{-1}[13]). This mismatch can result in tensile stresses within the material. In addition, the temperature gradient of EBFZM equipment has been calculated to be 350–500 K/cm[14], the high temperature gradient may easily induce stresses as well[15]. Therefore, the high strains are the most probable reasons of the $TaSi_2$ fibers bending if a high speed of crystallization is applied (R=6.0 mm/min). It is well known that convection is inevitable during the directional solidification. Therefore, the fiber is curved when it grows from the liquid, the convection and the high strains make the fiber curved. Further investigation of the formation mechanism of the curved $TaSi_2$ fiber is still needed.

3.2 Mechanical properties

All nanoindentation analyses are based on the Oliver and Pharr method[16]. The hardness (H) is determined from the peak load ($P_{\rm max}$) and the projected area of contact $A_{\rm c}$, as shown in the following equation

$$H = \frac{P_{\rm max}}{A_{\rm c}} \tag{1}$$

The reduced elastic modulus ($E_{\rm r}$) can be obtained by the following equation:

$$E_{\rm r} = S(\pi)^{0.5}/2(A_{\rm c})^{0.5} \tag{2}$$

where S is the slope of unloading curve, $A_{\rm c}$ is the projected area of contact. The area function for a perfect Berkovich tip is given as[17]

$$A_{\rm c} = 24.5h_{\rm c}^2 \tag{3}$$

The contact depth $h_{\rm c}$ is expressed as

$$h_{\rm c} = h_{\rm t} - \varepsilon\frac{P_{\rm max}}{d_{\rm p}/d_{\rm h}} \tag{4}$$

where dP/dh is stiffness, *i.e.* derivative of the power law fit to the unloading curve and evaluated at the maximum load, $P_{\rm max}$ is the maximum load, $h_{\rm t}$ is the maximum depth. ε is the tip shape constant which is equal to 0.72 for a conical tip. The load-displacement curves of the $TaSi_2$ fibers are shown in Fig. 2.

In Fig. 2, it is evident that the recovery of the straight $TaSi_2$ fiber is much slow compared with that of the bended $TaSi_2$ fiber. This demonstrates that the straight fiber undergoes plastic deformation, whereas the bended one undergoes the elastic-plastic deformation. In the present experiment, both the loading and unloading curves are continuous and consistent. Any discontinuities which would have indicated interfacial debonding, cracking, pop-in or pop-out during the test do not appear. As shown in Fig. 2, the maximum indentation depth of the bended $TaSi_2$ fiber is smaller than that of the straight $TaSi_2$ fiber.