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DISLOCATIONS AND MECHANICAL PROPERTIES OF
SINGLE CRYSTAL NIOBIUM DISILICIDE

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DISLOCATIONS AND MECHANICAL PROPERTIES OF SINGLE CRYSTAL NbSi_2

Proceedings

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NIOBIUM

DISILICIDE

Abstract

The mechanical properties of single crystal NbSi_2 have been investigated along [0001] and 45° from [0001] toward $[11\bar{2}0]$ using a Nikon QM-2 hot hardness tester from room temperature to 900°C in vacuum. The hardness along [0001] increases from room temperature to 300°C followed by a sharp decrease in hardness with temperature which is accompanied by a large uplift observed surrounding the indentations. Dislocations surrounding the indentations at room temperature and 300°C were analyzed using techniques in transmission electron microscopy to find slip by $\langle 10\bar{1}0 \rangle$ dislocations at room temperature with a change in the active slip systems at 300°C . The hardness along a direction 45° from [0001] toward $[11\bar{2}0]$ sharply decreases with increasing temperature above room temperature. Coarse slip lines surround the indentations referring to slip on the basal plane.

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Introduction

Because of growing interest in using silicides as high temperature structural materials, the mechanical properties of single crystal niobium disilicide are being investigated. NbSi_2 is an attractive high temperature material because of its low density, 5.704 g/cc, and high melting point, 1920°C. NbSi_2 has the C40 hexagonal crystal structure with $c=6.59\text{\AA}$ and $a=4.79\text{\AA}$ and c/a ratio of 1.374, space group, $P6/222$ (see Fig. 1a). Fig. 1b illustrating the basal plane in NbSi_2 shows the shortest lattice direction to be $1/3\langle 11\bar{2}0 \rangle$, 4.79Å. The next shortest lattice translation in the basal plane is $\langle 10\bar{1}0 \rangle$, 8.29Å, which can break up into three collinear $1/3\langle 10\bar{1}0 \rangle$ partials, 2.76Å, separated by APB's. The shortest lattice direction with some c -axis component is $[0001]$, 6.59Å followed by $1/3\langle 11\bar{2}3 \rangle$, 8.15Å.

Some research has already been performed on the mechanical properties of single crystal NbSi_2 , although this research was limited to deformation along an orientation approximately 45° from $[0001]$ toward $[11\bar{2}0]$ [1]. Compression ductility was observed at temperatures down to room temperature and a yield anomaly was observed around 1350°C. Slip traces referring to basal slip were observed after deformation.

This study looks at the mechanical properties of single crystal NbSi_2 along $[0001]$ and 45° from $[0001]$ toward $[11\bar{2}0]$ from room temperature to 1000°C using a hot hardness tester. Dislocations around indents are characterized to determine the active slip systems around indentations made at specific temperatures.

Figure 1 Schematic showing (a) the crystal structure of NbSi_2 and (b) the $[0001]$ plane of NbSi_2 .

Experimental Procedure

Single crystal NbSi₂ was grown by J. Garrett using the Czochralski growth technique at McMaster University. Single crystals were oriented using a Laue back reflection X-ray machine along [0001] and 45° from [0001] toward $\langle 11\bar{2}0 \rangle$. Foils were ion milled to electron transparency to analyze the initial dislocation density in the starting material. Samples were cut into parallelepipeds approximately 5x5x10 mm in size. One side was polished down to 1μm diamond paste. A Nikon QM-2 hot hardness tester was used to determine the hardness of single crystal NbSi₂ in vacuum from room temperature to 1000°C by measuring five indentations at each temperature. Slip traces and cracks around indentations were observed using a JEOL 6300FVX field emission scanning electron microscope at 25 kV. Thin foils were back thinned around indentations using a Gatan ion mill. Dislocations around indentations were analyzed by performing g•b and trace analyses with a Philips CM30 TEM.

Results

45° from [0001] toward $[11\bar{2}0]$

The hardness measured along 45° from [0001] toward $[11\bar{2}0]$ is shown in Fig. 2. The hardness sharply decreases with increasing temperature from room temperature up to 900°C. Hardness was not measured above 900°C as the indenter reacted with the specimen. Coarse slip lines on the basal plane and cracking were observed around all indentations at all temperatures as shown in Fig. 3 for a typical indentation performed at 900°C. Knoop indentation was also performed at room temperature with the long direction of the indenter oriented parallel to $[1\bar{1}00]$ and perpendicular to $[1\bar{1}00]$. No slip lines on the basal plane or cracks around the indentation were observed when the long direction of the indenter was oriented perpendicular to $[10\bar{1}0]$ while coarse slip lines on the basal plane and cracks were observed around indentations performed with the indenter oriented parallel to $[1\bar{1}00]$ (see Fig. 4). The hardness was highest when the indenter was oriented parallel to $[1\bar{1}00]$ (683 ± 5 kg/mm²) and softest (642 ± 16 kg/mm²) when indented perpendicular to $[1\bar{1}00]$.

[0001]

The variation of hardness along [0001] from room temperature to 700°C is shown in Fig. 2. The hardness anomalously increases with temperature up from 100 to 300°C followed by a decrease with temperature up to 700°C. The anomalous increase in hardness observed starting at 300°C is accompanied a marked change in the appearance of the hardness indentation starting at

Figure 2 Graph showing hardness vs. temperature for single crystal NbSi_2 along two different orientations.

Figure 3 Optical micrograph showing an indentation performed at 900°C along a direction 45° from [0001] toward $[11\bar{2}0]$

Figure 4 Optical micrographs showing Knoop indentations performed in a direction 45° from [0001] toward $\langle 11\bar{2}0 \rangle$ with the long axis of the indenter (a) parallel to $[1\bar{1}00]$ and (b) perpendicular to $[1\bar{1}00]$.

200°C. Fig. 5 shows an overall optical micrograph of the hardness indentations from room temperature to 500°C. There is an obvious change in the appearance of the uplift around the indent at 200°C from a gradual uplift at temperatures below 200°C to a distinct hexagonal shaped uplift at 200°C and above.

This anomaly was studied further using scanning electron microscopy. The results reveal a difference in cracking and the appearance of slip lines from observing indents performed below and above 300°C (see figure 6a and b). Cracking was random around the indents below 100°C while it changed to cracking inside the indentation which was parallel to the basal plane at 100°C and above. Slip traces were observed in the bottom of indents performed at room temperature but not at any other temperatures. The slip traces were observed in a triangular pattern.

To analyze the deformation mechanisms active around indentations at room temperature and 300°C, TEM foils were back-thinned around indents performed at room temperature and 300°C. The g.b analysis in Fig. 7a-d reveals two sets of dislocations around an indentation performed at room temperature. The first set (labeled A in Fig. 7a) go out of contrast when imaged with $g=(1\bar{2}11)$ and $g=(1\bar{2}10)$. Therefore, they have $[10\bar{1}0]$ Burgers vectors. The second set (labeled B in Fig. 7a) go out of contrast with $g=(11\bar{2}0)$ and $g=(11\bar{2}1)$. Therefore, they have $[1\bar{1}00]$ Burgers vectors. Slip trace analyses on these sets of dislocations revealed that dislocations labeled A have a $[10\bar{1}0]$ line direction while those labeled B have a $[1\bar{1}00]$ line direction making them pure screw dislocations

Dislocations around indentations performed at 300°C were also observed. The dislocations were observed using many different g-vectors, although they were only clearly invisible when imaged with $g=(01\bar{1}0)$ (labeled A in Fig. 8a) and $g=(10\bar{1}0)$ (labeled B in Fig. 8a). The

Figure 5 Optical micrograph showing indentations performed along $[0001]$ from room temperature to 500°C.

Figure 6 Scanning electron micrograph showing indentations performed along $[0001]$ at (a) room temperature and (b) 200°C .

Figure 7 Bright field transmission electron micrographs showing dislocations around an indentation performed at room temperature along $[0001]$. Imaged with (a) $g=(1\bar{2}11)$, (b) $g=(1\bar{2}10)$, (c) $g=(11\bar{2}0)$ and (d) $g=(11\bar{2}1)$.

Figure 8 Bright field transmission electron micrographs showing dislocations around an indentation performed at 300°C along [0001]. Imaged with (a) $g = (01\bar{1}0)$ and (b) $g = (10\bar{1}0)$.

dislocations were clearly visible when imaged with $\langle 11\bar{2}0 \rangle$ g-vectors ruling out the possibility of the dislocations having a $\langle 10\bar{1}0 \rangle$ Burgers vector.

Discussion

45° from [0001] toward $[11\bar{2}0]$

The hardness results along a direction 45° from [0001] toward $[11\bar{2}0]$ show the ease of basal slip in NbSi₂. The coarse slip bands on the [0001] plane reveal that nucleation of slip is difficult while dislocation glide on the basal plane is easy. This is not surprising because the basal plane is the densest plane in NbSi₂. The anisotropy in hardness observed from the Knoop indentations also confirms the ease of slip on the basal plane, because the hardness was higher when no basal slip traces were observed and softer when numerous basal slip traces were observed. Future work will determine the active slip systems around Knoop indentations in each case using transmission electron microscopy. It is surprising that a high degree of cracking accompanies indentations exhibiting basal slip. This may result from high stresses built up between the coarse slip bands.

In comparing our hardness results along a direction 45° from [0001] toward $[11\bar{2}0]$ to compression results measured by Umakoshi et al.[1], one can see a strong agreement as they observe basal slip after deformation at all temperatures. Future work will investigate the hardness at higher temperatures up to 1400°C where they reported a yield anomaly.

[0001]

An interesting anomaly in the hardness vs. temperature curve is observed for deformation in the [0001] orientation. This anomaly is accompanied by a change in the uplift surrounding the

indents from gradual to a distinct uplift with a hexagonal shape. The change in cracking from a random crack pattern surrounding the indent to cracking within the indent on the basal plane suggests that the uplift is associated with cracking on the basal plane.

The results of the dislocation analyses show that there is a change in slip system associated with the anomaly. Slip at lower temperatures occurs by $\langle 10\bar{1}0 \rangle$ Burgers vectors while slip at higher temperatures occurs by either $\frac{1}{3}\langle 11\bar{2}3 \rangle$ or $\frac{1}{3}\langle 11\bar{2}0 \rangle$ Burgers vectors. Further TEM analyses will resolve this active slip system at higher temperatures.

Conclusions

- 1) The hardness of single crystal NbSi_2 has been determined from room temperature to 900°C along a direction 45° from $[0001]$ toward $[11\bar{2}0]$ and along $[0001]$.
- 2) Basal slip is observed around all indentations performed in a direction 45° from $[0001]$ toward $[11\bar{2}0]$.
- 3) The hardness measured along $[0001]$ anomalously increases with temperature from 100°C peaking at 300°C .
- 4) Dislocations around indents performed at room temperature along $[0001]$ have $[10\bar{1}0]$ Burgers vectors while a change in slip systems is observed at 300°C .
- 5) Cracking on the basal plane is observed within indents performed along $[0001]$ at 100°C and above.

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Reference

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