

## **Final Report for**

**Twinning in Laves Phases by Synchroshear:  
Atomic Mechanisms and Compositional Control**

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### Abstract

Compression tests between 4.2 and 1273 K, compositional variation and conventional transmission electron microscopy were used to investigate the deformation and twinning behavior of C15 HfV<sub>2</sub>+Nb - based Laves phase alloys. We chose two phase C15/bcc alloys to improve the ambient temperature ductility which has not been found in the single phase Laves intermetallic compound. Transmission electron microscopy revealed that substantial mechanical twinning takes place in the C15 Laves phase matrix at room temperature and 77 K. A deep minimum in the flow stress, with a drop of nearly 500 MPa, appears at around 77 K in the C15/bcc two-phase alloy with C15 matrix. Since no such anomaly is seen in the bcc phase, we believe that the cause of this can be attributed to mechanical twinning in the C15 Laves phase. Twin bands observed in the C15 matrix of deformed samples at both 298 K and 77 K can be classified into three categories by their thicknesses; coarse twin bands about 10 nm to several hundreds nm thick, fine twin bands around 3 to 10 nm thick, and ultra fine twin bands with average thickness of 1.5 nm. A high density of ultra-fine twin bands is the characteristic feature of twinning in the C15 matrix. They belong to the  $\langle 112 \rangle (111)$  twinning system and commonly intersect with each other.

### Introduction

Structural materials for elevated temperature application require minimum, highly reliable toughness values. Generally, toughness is governed by two conflicting properties of a material, strength and ductility. Intermetallic compounds having high melting points and superior elevated temperature strength can be good candidate materials only if they have sufficient ambient temperature ductility. Extensive research has been carried out on intermetallic compounds with simple crystal structures, based on the bcc and fcc structures found in pure metals. This was considered to be a route by which to achieve substantial plastic deformability, because insufficient low temperature ductility is the largest drawback of intermetallic compounds. An examination of phase diagrams reveals many "exotic" intermetallic compounds that have complex crystal structures. The Laves phases represent a huge group of these binary intermetallic compounds, ~27% of all known binary compounds [1,2]. These phases have reasonably simple crystal structures categorized as derivatives of fcc and hcp. The C15 Laves phase, for example HfV<sub>2</sub>, has a crystal structure that is derivative of the diamond structure, i.e., based on fcc. Studies of the room temperature mechanical properties of the Laves phases have shown that the toughnesses of C15 single-phase alloys are very limited. However, two strategies have been identified to improve the toughness of these alloys, addition of a ternary element and alloying with a ductile phase.

In spite of its relatively simple cubic crystal structure, the binary C15 Laves phase HfV<sub>2</sub> shows almost no plasticity at low temperature. It is easily imagined that dislocation slip cannot operate in the Laves phases because of their complex ordered crystal structures and tight spacing of close-packed planes. The addition of Nb is known to improve the plastic



deformability of C15 HfV<sub>2</sub> through mechanical twinning [3,4], however, polycrystalline HfV<sub>2</sub>+Nb single phase alloys are still extremely brittle [4]. Two-phase alloys consisting of the C15 HfV<sub>2</sub>+Nb and the bcc (V-Nb) solid solution exhibit some ductility in compression but not in tension at ambient temperature, even though the matrix of the alloy is the brittle C15 phase.

Over the course of this research project, compression tests were performed on the 14Hf-64V-22Nb alloy, C15/bcc two-phase with the C15 matrix, over a wide temperature range from room temperature to 1273 K [4,5]. This alloy exhibits three types of mechanical behavior over this range of temperature. From room temperature to 600 K, the alloy exhibits some compressive ductility to about 3 to 4 % plastic strain. Mechanical twinning of <112>(111) type, a typical fcc type, was reported as the major deformation mode in the C15 Laves phase, and the mechanical behavior is strain rate insensitive in this temperature range. At intermediate temperatures between 600 K and 1000 K, the alloy exhibits little plastic deformability. This minimum in the ductility seems to be due to the lack of mechanical twinning in the C15 matrix. At elevated temperatures exceeding 1100 K, the alloy shows excellent ductility with virtually unlimited plastic strain. Dislocation slip with a burgers vector  $b=1/2\langle 110 \rangle$  is the deformation mechanism in the C15 Laves phase [4].

Although brittle at low temperatures, the 14Hf-64V-22Nb alloy undergoes a brittle-to-ductile transition (BDT) at around 1100 K ( $\sim 0.65T_m$ ). It is interesting to note that the BDT temperature is close to the temperature of the peak in the yield stress; the well-known anomalous temperature dependence of the flow stress found in many intermetallic compounds. This behavior indicates that the loss of ductility at intermediate temperatures could be attributed to the cessation of dislocation slip due to a lack of mobility governed by thermal activation. If this is true, then neither dislocation slip nor mechanical twinning operate within the HfV<sub>2</sub>+Nb Laves phase within the intermediate temperature region. This suggests that the cessation of twinning is not related to the initiation of dislocation slip and questions the popularly held view that twinning stops when dislocation slip commences.

The C15 Laves phase is an AB<sub>2</sub> type compound of which the unit cell is shown in Fig. 1(a). This is a so-called topologically close-packed (TCP) structure. The C15 ordered crystal structure can be related to both the fcc and the diamond structure in the following way: The large Hf atoms are situated exactly on the diamond structure lattice points and hence have a tetrahedral stacking sequence of (111) planes given by ... $\alpha\alpha\beta\beta\gamma\gamma$ ... The small V atoms are arranged in close-packed layers containing ordered vacancies between the layers of Hf atoms such that the large Hf atoms protrude into the periodic vacant positions in the V layers. As shown in Fig. 1(b). The vacant positions in the V layer comprise a Kagome net. Such V layers are called A, B, or C layers. Thus the two atomic species are arranged in such a way that individual (111) planes contain only one kind of atom. In a V layer between  $\alpha$  and  $\beta$  (or  $\beta$  and  $\gamma$ , or  $\gamma$  and  $\alpha$ ) Hf layers, three-fourths of the V sites are vacant, as shown in Fig. 1(c). Such layers are called a, b, or c layers. Thus the (111) plane stacking in the C15 structure consists of a twelve-layer sequence of ... $\alpha A\alpha c\beta B\beta a\gamma C\gamma b\alpha$ ..., and can be considered to be "fcc-like" with the  $\alpha A\alpha c$ , the  $\beta B\beta a$ , and the  $\gamma C\gamma b$  four-plane stacks in the C15 phase corresponding to the a, b,



and c layers in the ...abc... stacking sequence of the fcc crystal structure.

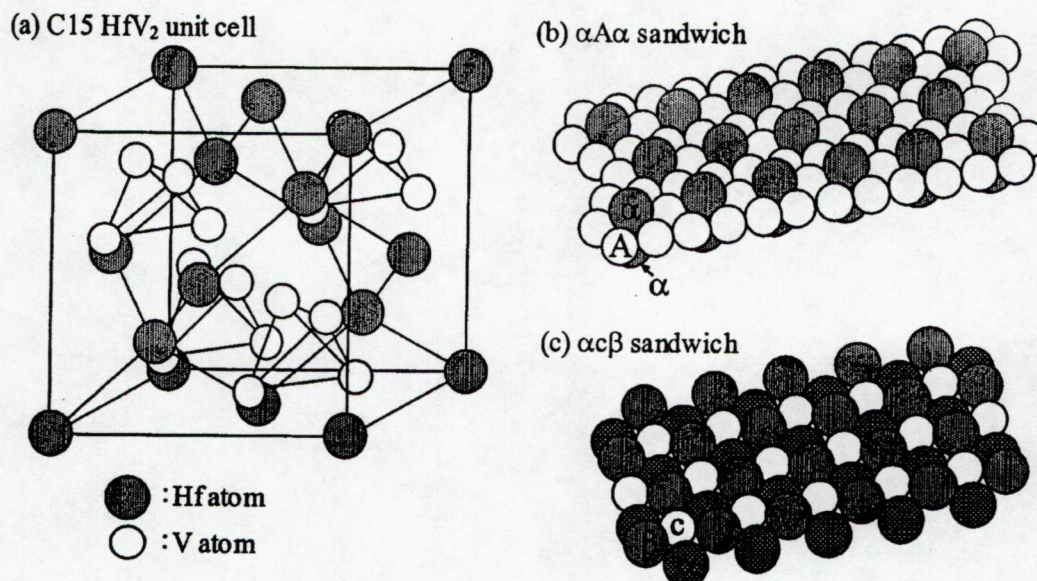


Fig. 1 C15 Laves phase crystal structure; (a) the unit cell, and two characteristic stacking sequence of (111) planes (b) " $\alpha A \alpha$  sandwich" and (c) " $\alpha c \beta$  sandwich."

### Experimental Procedures

Alloys were prepared by arc melting in an argon gas atmosphere using high purity raw materials, purity of 99.9% Hf, 99.7% V, and 99.9% Nb. As-cast ingots were annealed at 1473 K for 72 hs in an argon gas flow. Microstructures were observed using back scattered electron images in a JEOL 6400 scanning electron microscope (SEM).

Compression tests were conducted at temperatures ranging from room temperature down to 4.2 K. Compression tests were performed on an Instron Model 1125 universal mechanical testing machine equipped with a double-dewar for immersion within a constant temperature bath. Test temperature was monitored by carbon resistors attached to the apparatus above and below the specimen and with a thermocouple on the specimen. An appropriate foam casing was prepared to help maintain low temperatures especially for the test at 4.2 K. Compression test specimens of dimensions  $3 \times 3 \times 6$  and/or  $2.5 \times 2.5 \times 5 \text{ mm}^3$  were cut from the as-homogenized ingots by electron-discharge machining (EDM). Initial strain rates were 7.1 and/or  $8.6 \times 10^{-4} \text{ s}^{-1}$ , for the different sized specimens, respectively.

The microstructures of deformed and as-homogenized materials were observed using a JEOL 4000EX transmission electron microscope (TEM) operated at an accelerating voltage of 400 kV. TEM specimens were prepared from the as-homogenized material and from deformed materials after a compressive deformation of about 2 % plastic strain. Thin disks were cut from the material using EDM. As-cut disks were mechanically polished to about  $90 \mu\text{m}$  and dimpled on one side until the center of the dimple was approximately  $30 \mu\text{m}$  thick. Final perforation of the disks was achieved using a GATAN DuoMill conventional argon ion mill operated at 77 K or a GATAN Precision Ion Polishing System (PIPS). The specimen-Argon ion gun angle in the



PIPS was 4 degrees and a beam energy of 5 keV was used.

### Experimental Results

Figure 2 shows the isothermal section of the Hf-V-Nb ternary system at 1273 K, which was evaluated by Chu et al. [4,8]. The wide composition range of the C15/bcc two-phase field allows us to select a wide range of volume fraction ratios of the constituent phases with different Nb contents. The chemical compositions of the alloys on which compression tests were conducted in the current study are shown on the diagram. In the present work, the solubility limit of Hf in the (V, Nb) solid solution was found to possibly be less than that reported in the prior work [4,8]. Back-scattered electron images of the C15/bcc two-phase microstructures for alloys MP1 and MP2 are shown in Fig. 3. The white contrast is the C15 Laves phase  $\text{HfV}_2\text{+Nb}$ , and the dark contrast, the bcc solid solution (V, Nb). Alloy MP1 has a C15 matrix and C15 small precipitates in the second phase bcc, while alloy MP2 has a bcc matrix instead of the C15. Note that alloys MP1 and MP2 share the same composition tie line but have different volume fractions of the constituent phases. Alloys MP1, MP3 and MP4 have almost the same microstructure, i.e. the volume fractions are constant, in an effort to observe the effect of differences in Nb content on the deformation behavior of the alloy.

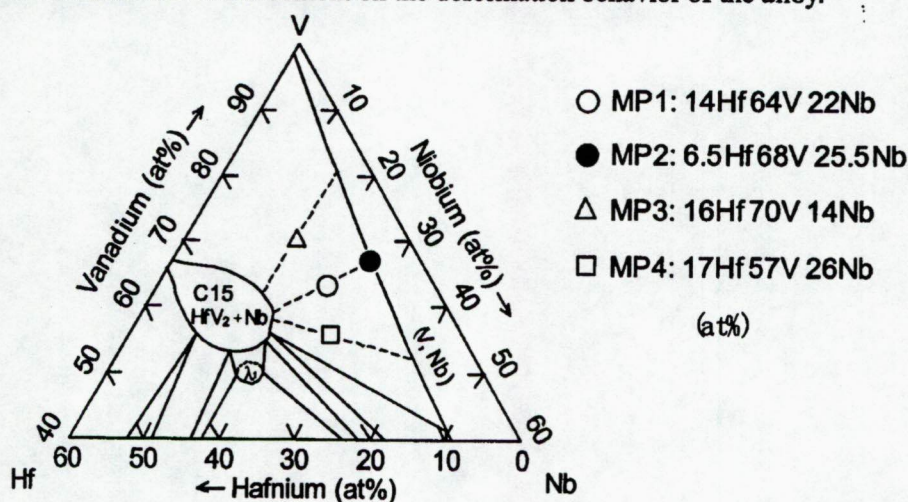


Fig. 2 The isothermal section of the Hf-V-Nb ternary system at 1273 K evaluated by F. Chu. Chemical compositions of present alloys are shown as well.

The temperature dependence of the 0.2% compressive flow stress of the C15/bcc two-phase alloys MP1 through MP3 is shown in Fig. 4. Note that this is just a portion of the overall temperature range investigated, as described in the introduction. The flow stress of alloy MP1, which has a C15 matrix, gradually increases as the temperature is decreased but then a



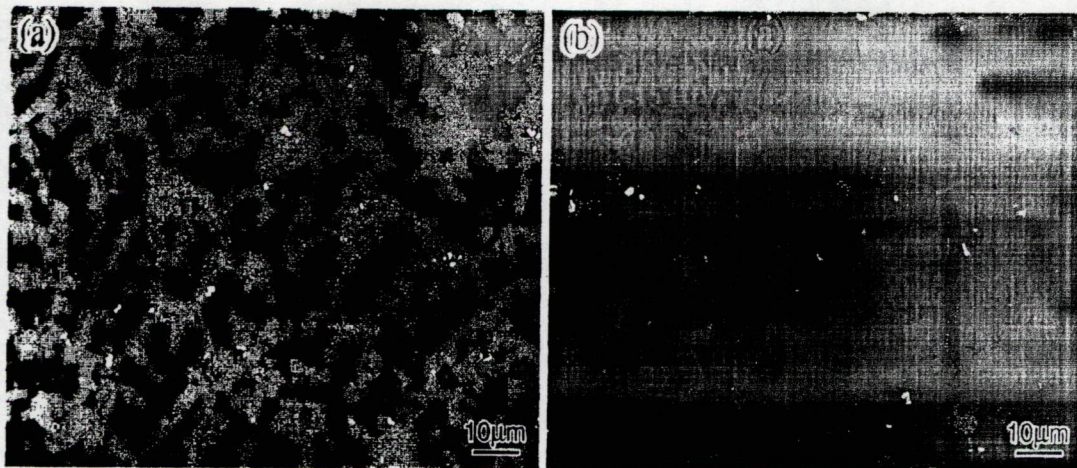


Fig 3 Back scattering electron images of the C15/bcc two-phase alloys; (a) 14Hf 64V 22Nb alloy with the C15 matrix and (b) 6.5Hf 68V 25.5Nb alloy with the bcc matrix.

sharp minimum appears at about 77 K. Further cooling results in a rapid increase near 4.2 K and a total loss of ductility. The curve for alloy MP3, which has almost the same microstructure as alloy MP1 but contains less Nb, appears to show a shoulder in this temperature range. Thus reducing the Nb content appears to lower the strength level of the material and to reduce the anomaly in the low temperature mechanical properties. Contrary to our expectation that increases in Nb improves ductility, alloy MP4 shows no plasticity and fractures within the elastic region. Alloy MP2, which has a bcc (V, Nb) matrix instead of the C15 matrix, shows no such anomaly in the mechanical behavior. The behavior of this bcc matrix alloy is somewhat unusual because we would expect to see a more rapid and monotonic strengthening as the temperature is reduced.

Analysis of the deformed microstructure in the TEM confirmed that the Laves phase deformed by mechanical twinning. The results indicate that twinning becomes much easier around 77 K. The comparison between the behavior of the bcc matrix alloy and the C15 matrix alloy indicates that the large dip at 77 K is a result of changes in the C15 phase, not the bcc phase. The fact that the mechanical anomaly has an obvious dependence on Nb content supports this idea as well, since the addition of Nb is known to enable and enhance mechanical twinning in the C15 Laves phase. The results obtained on alloy MP4 implies that too much Nb content might induce thermal twinning and/or stacking faults that could then resist the mechanical twinning.

Transmission electron microscopy, using imaging and diffraction analysis, was performed to investigate the twinning behavior in the C15 Laves phase  $\text{HfV}_2\text{+Nb}$  focusing on the role of mechanical twinning on the plastic deformation of the C15/bcc two-phase material. Typical bright field images of twin bands observed in the C15 Laves phase are shown in Fig. 5(a) through (d). Twin bands seen in the C15 matrix of samples deformed at both 298 K and 77 K can be classified into three categories by their thickness; i) coarse twin bands ranging from about 10 nm to several hundreds nm thickness shown in Fig. 5(a), ii) fine twin bands around 3 to 10 nm thickness shown in Fig. 5(b), and iii) ultra fine twin bands with average size of 1.5 nm



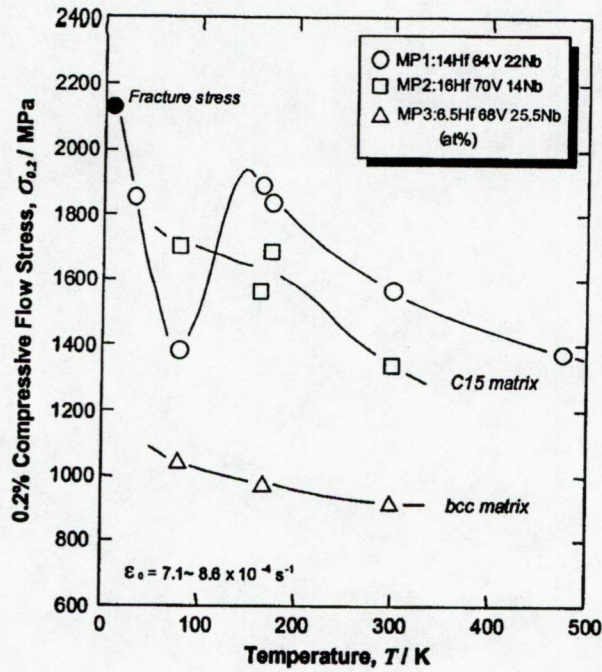


Fig. 4 The temperature dependence of 0.2% compressive flow stress for the C15/bcc two-phase alloys, MP1, MP2 and MP3.

thickness shown in Fig. 5(c). All three types of twin bands are seen in the as-homogenized material as well, though the populations, except for the population of the coarse twin bands, is qualitatively much lower than in the deformed material. Twin bands can be thermally introduced during cooling from high temperature, i.e., after the homogenization heat treatment at 1473 K for 72 hs. Thermal stress due to the difference of thermal expansion coefficients between the C15 and the bcc solid solution may also cause twinning at the interfaces between the C15 and the bcc phases. The addition of Nb is thought to lower the stacking fault energy in the C15 HfV<sub>2</sub> phase so that the formation of stacking faults and twin bands may be promoted with increasing Nb content. Lowering the stacking fault energy would ease the formation of twins, either thermally driven or stress driven.

To see if the strength decrease at 77 K is the result of a change in deformation mechanism, the deformation structure of a sample deformed at room temperature was compared to that of a sample deformed at 77 K as we reported previously [9]. The three distinctive morphological constituents were examined, the matrix C15 Laves phase, the fine C15 precipitates within the bcc solid solution and the bcc solid solution. The structures appear to be qualitatively the same at both deformation temperatures, viz., the C15 Laves phase deforms by profuse twinning, and twin spots are readily visible in the diffraction patterns. In the bcc phase, substantial dislocation tangles are observed, which indicates that ordinary dislocation slip is the deformation mode in the bcc at this temperature range.



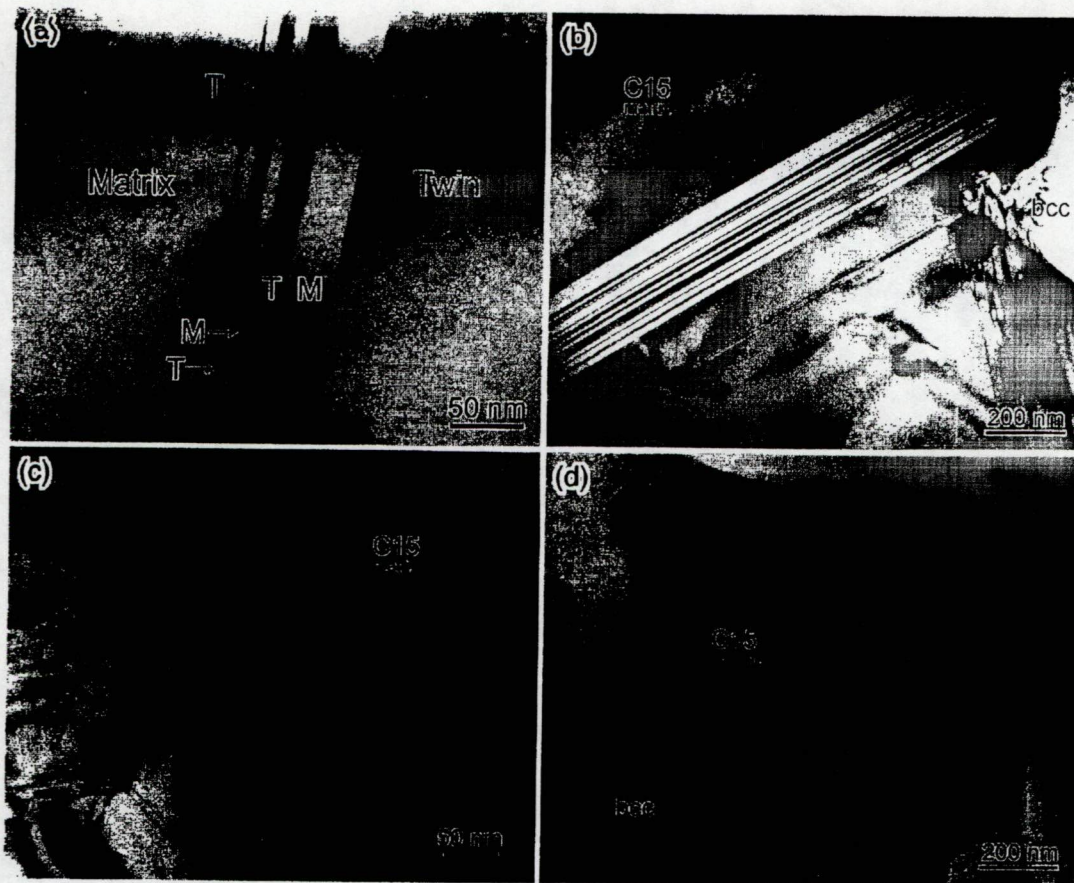


Fig. 5 Bright field images of typical twin bands observed in the C15 Laves phase  $\text{HfV}_2\text{Nb}$ . (a) coarse twin bands, (b) fine twin bands and (c) ultra-fine twin bands formed in the C15 matrix of the C15/bcc two-phase alloy, and (d) fine twin bands in the Ca15 small precipitate within the second phase bcc (V, Nb).

An example of coarse twin bands is shown in Fig. 5(a). It is interesting that the left portion and the right portion in the same C15 grain have the matrix-twin relationship. It is obvious that this type of coarse twin band is unlikely to be produced by stress-induced plastic deformation, because the size of the twin band is far too thick to shear. Therefore, these coarse twin bands could be attributed to the result of solidification leaving the matrix-twin relationship.

Another possible mechanism for the production of matrix-twin type coarse twin bands is the C14 – C15 phase transformation. In the Hf-V-Nb ternary system, a C14 phase field has been found on the more Nb-rich side of the C15 phase field in the 1273 K isotherm as shown in Fig. 2. This is consistent with the possibility that Nb reduces the stacking fault energy of the cubic  $\text{HfV}_2$  Laves phase. However, no further information is available for temperatures higher than 1273 K. In other alloy systems, it is common for the hexagonal C14 phase to be the high temperature phase transforming to the cubic C15 phase as the temperature is reduced. The existence of a high-temperature C14 phase at the compositions studied in the present work cannot be discounted.



Fine twin bands are seen in both the C15 matrix, Fig. 5(b), and in the fine C15 precipitates within the bcc (V, Nb) solid solution phase, Fig. 5(d). Many of fine twin bands nucleate at the C15/bcc interface and at grain boundaries of the C15, where a stress concentration would be expected to exist, as seen in Fig. 5(b). Fine twin bands are presumably formed under mechanical stress during the compression test at both 298 K and 77 K. It is expected that the fine twin bands can provide a substantial plastic strain.

A characteristic feature of fine twin bands is that they are comprised of many thin twin plates. The spacings between the twin plates are small. In the case of the fine twin bands formed in the fine C15 precipitates, the total width of a group of fine twin bands sometimes becomes considerably thick as shown in Fig. 5(d).

A high density of ultra-fine twin bands are found, but only in the C15 matrix, as shown in Fig. 5(c). As mentioned above, the ultra-fine twin bands are also observed in an as-homogenized specimen. For the finest ultra-fine twin bands, it is difficult to distinguish them from a stacking fault. In fact, a single layer twin is crystallographically identical to an intrinsic stacking fault; a double layer twin is crystallographically identical to an extrinsic stacking fault. The concept of a "mechanical stacking fault" vs. a "mechanical twinning" has been reported for the  $L_{12}$   $Ni_3Al$  intermetallic compound in the literature [10-13]. A thickness of around 1.5 nm corresponds to the shear of a two or three layered twin in the C15 structure. The population of ultra-fine twin bands seems to be larger in the sample deformed at 77 K than in the one deformed at room temperature, although this is a qualitative observation using TEM.

A dark field image and bright field images of ultra-fine twin bands observed in the C15 matrix are shown in Fig. 6 (a) and (b). Each index in the figure represents the shear plane and shear vector, evaluated using the invisibility criteria,  $g \cdot R = 0$  where  $g$  is the operating reflection and  $R$  is the atomic displacement across the twin (stacking fault), which is obtained using several two-beam conditions. All the twin bands analyzed belong to the  $\langle 112 \rangle \{111\}$  twinning system.

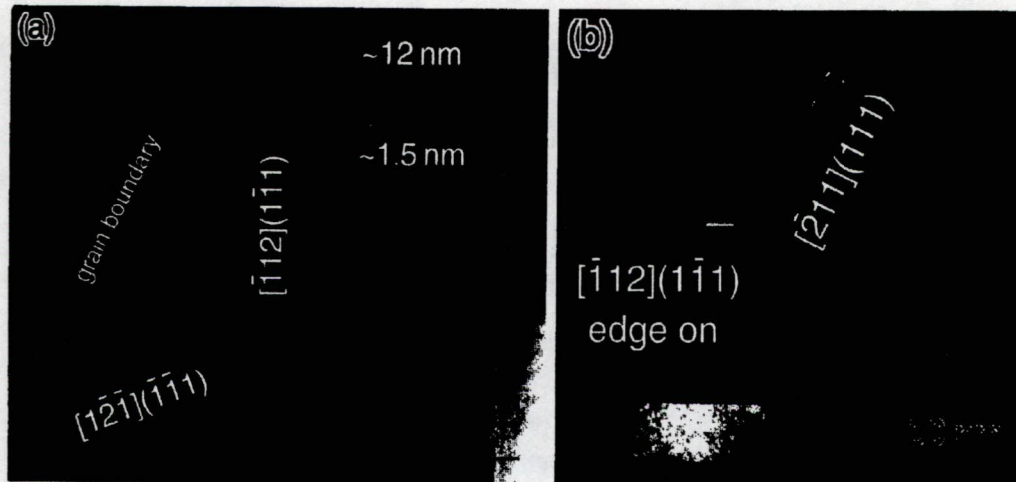


Fig. 6 A bright field image (a) and a dark field image (b) showing ultra-fine twin bands observed in the C15 matrix of the C15/bcc two-phase alloy deformed at 77 K in compression.



As seen in Fig. 5(c) and Fig. 6, one characteristic feature of ultra-fine twin bands is that they intersect each other, unlike the thicker twin bands. This may be evidence that the ultra-fine twin bands make large contribution to the plastic deformation of the material. The displacement vector at the intersection of two twin bands can be determined by the addition or subtraction of the individual shear vectors. If a twin band propagates through by changing its direction at the intersection, a  $[011]$  type displacement vector is left at the intersection. In the case that two twinning bands meet with or collide at the intersection, a  $[323]$  type displacement vector is produced.

### Discussion

The C15 Laves phase structure (cf. Section 1.2) can be conceptually divided into two different three-plane stacks, both composed of a central V layer with adjacent Hf layers on both sides. These "sandwiches" are then of the types  $\alpha A\alpha$ , ( $\beta\beta\beta$ ,  $\gamma C\gamma$ ) (Fig. 1(b)) and  $\alpha c\beta$ , ( $\beta a\gamma$ ,  $\gamma b\alpha$ ) (Fig. 1(c)). The Hf-Hf spacing perpendicular to the atomic planes in the  $\alpha A\alpha$ , ( $\beta\beta\beta$ ,  $\gamma C\gamma$ ) sandwiches is three times larger than that in the  $\alpha c\beta$  ( $\beta a\gamma$ ,  $\gamma b\alpha$ ) sandwiches. However, since the Hf atoms are positioned at vacant sites of the V atomic plane in  $\alpha A\alpha$ : ( $\beta\beta\beta$ ,  $\gamma C\gamma$ ) sandwiches, these sandwiches are quite rigid against in-plane shear.

Prior work has shown that the twinning plane is the high density V plane, i.e., the A, B, or C plane [4,14]. With the  $\alpha A\alpha$  ( $\beta\beta\beta$ ,  $\gamma C\gamma$ ) sandwiches remaining intact, the twinning shear must be concentrated in the  $\alpha c\beta$  ( $\beta a\gamma$ ,  $\gamma b\alpha$ ) sandwiches. In general, in order for a net  $1/6\langle 112 \rangle$  twinning shear to occur in these sandwiches without producing steric conflicts between the atoms, either a homogeneous shear accompanied by diffusive atomic shuffles or a synchronized heterogeneous shear distributed over two atomic planes must occur. The observation of deformation twinning at 77 K in the present and prior work indicates that it is unlikely to be the result of atomic shuffles by a thermally activated mechanism, unless they can occur with a very small activation energy.

Based on the evidence to date, a twinning mechanism involving synchronized shears, termed synchroshear, is the most likely twinning mechanism in the cubic Laves phase. In this mechanism one layer of the sandwich shears along the direction of one Shockley partial dislocation while the next layer simultaneously shears in the direction of another Shockley partial such that the sum of the two is the necessary twinning shear. Thus, for example, within the (111)  $\alpha c\beta$  sandwich, the c V plane undergoes  $1/6[-12-1]$  shear with respect to the  $\alpha$  Hf plane and the  $\beta$  Hf plane undergoes  $1/6[2-1-1]$  shear with respect to the c V plane, producing a net shear of  $1/6[11-2]$  across the entire  $\alpha c\beta$  sandwich and producing no steric conflicts.

The cooperative simultaneous shear of both large and small atoms is one important feature of the synchroshear mechanism in the C15 Laves phase. The intervening material moves as a rigid block with no other shears. In the ternary C15 alloy  $\text{HfV}_2 + \text{Nb}$ , Hf and V occupy only their own sites (sublattices) and Nb substitutes on both Hf and V sites in such a way as to satisfy stoichiometry. In alloys with higher V contents Nb substitutes mostly for Hf sites, effectively producing free volume within the deformable  $\alpha c\beta$  sandwiches, which results, we believe, in easier formation of twins in these alloys. The fact that twinning is profuse at 77



K in the C15 HfV<sub>2</sub>+Nb provides additional evidence for the syncroshear mechanism.

Moreover, the C15 HfV<sub>2</sub>+Nb is a near-perfect material to study "stress driven vs. stress and temperature driven" twinning because the temperature range for twinning is several hundred degrees below that for dislocation slip [4,5]. Reviewing our previous results described in the introduction section, mechanical twinning in C15 HfV<sub>2</sub>+Nb stops at a temperature around 673 K. At test temperatures between 298 K and 77 K, compressive ductility of the alloy is comparable, showing 2 to 3% plastic strain. Cooling down to 4.2 K results in the complete loss of ductility in the alloys studied in the present work. Consequently, this temperature dependence of the plasticity indicates that mechanical twinning in the C15 HfV<sub>2</sub>+Nb is both a stress and temperature driven phenomenon.

### Conclusions Drawn From This Program

The following conclusions are drawn from this program.

1. A large flow stress dip appears in the yield strength of certain HfV<sub>2</sub>+Nb C15 matrix alloys near 77 K. This anomaly is the result of increased ease and amounts of mechanical twinning in the C15 Laves phase.
2. The twin bands found in the C15 matrix of deformed samples can be classified into three categories by thickness; i) coarse twin bands ranging from about 10 nm to several hundreds nm thickness, ii) fine twin bands around 3 to 10 nm thickness, and iii) ultra fine twin bands with average size of 1.5 nm thickness.
3. A high density of ultra-fine twin bands which intersect with each other is a distinctive feature of twinning in the C15 matrix of deformed samples. Transmission electron microscopic analysis using invisible criterion revealed that ultra-fine twin bands belong to the <112>(111) type twinning system. The density of these ultra-fine bands appears to be higher at low temperatures.
4. The fact that twinning is profuse at 77K in the Laves phase provides additional evidence for the syncroshear twinning mechanism.
5. The increased propensity for twinning with Nb additions is consistent with expectations based on the syncroshear mechanism.

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