ULTRASONIC VELOCITY AND TEXTURE OF HIGH RRR NIOBIUM

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VITESSE ULTRASONORE ET TEXTURE DU NIOBIUM DE HAUT RRR

RÉSUMÉ: L'évaluation conventionnelle par des essais de traction, des propriétés mécaniques du niobium de RRR élevé, laminé en plaques, ont révélé un module d'Young apparent et une limite d'élasticité exceptionnellement bas. Ces observations ont motivé une série de mesures de vitesse ultrasonore, destinée à la détermination dynamique des modules élastiques. En fait, le module dynamique est situé dans le domaine normal pour tous les échantillons testés. Cependant, il apparaît une tendance à la croissance des vitesses de cisaillement pour les ondes de cisaillement se propageant à travers l'épaisseur de la plaque et polarisées dans la direction transverse de la plaque. Des analyses précises de texture cristallographique par EBSD ont montré un changement de texture subtil mais systématique qui peut expliquer la tendance observée. Il est important de noter que le changement de texture n'est pas observable par des mesures en surface utilisant la diffraction des rayons X mais qu'il faut sectionner les échantillons. Ainsi, les mesures de vitesse ultrasonore représentent un outil d'évaluation non destructive qui est extrêmement sensible aux variations fines de texture du niobium de RRR élevé. Finalement, il y a des variations liées au lot de métal qui sont couramment attribuées aux effets des impuretés telles que Ta et H.

ABSTRACT: Conventional assessments of the mechanical properties of rolled high RRR niobium plate material via tensile testing have revealed an unusually low *apparent* Young's moduli and yield strength in some annealed samples. These observations motivated a series of measurements of ultrasonic velocity, a dynamic assessment of the elastic moduli. In fact, the dynamic modulus is within the range of normal for all samples tested. However, there is a trend of increasing shear velocities for shear waves propagating through the sheet thickness and polarized in the sheet transverse direction. Careful analyses of the crystallographic texture using SEM-based electron backscattered diffraction (EBSD) have revealed a subtle, but systematic change in the texture, which can explain the trend. It is further important to note that the change in texture is not observable from surface measurements using X-ray diffraction, but requires sectioning of the samples. Thus, measurements of ultrasonic velocity represent a non-destructive evaluation tool which is extremely sensitive to subtle changes in the texture of high RRR niobium. Finally, there are material lot variations, which are currently attributed to the effects of impurities, such as Ta and H.

Superconducting radio frequency cavities used as accelerating structures in particle accelerators are made from high purity niobium with Residual Resistivity Ratio (RRR) values of greater than 250, corresponding to a thermal conductivity of approximately 60 W/m K at 4.2 K. The major impurities are tantalum and interstitially dissolved gases such as hydrogen, nitrogen, and oxygen in addition to carbon. The concentration of hydrogen, in particular, is important because

of its known connection with the so-called "Q-disease" (increased losses in conductivity by a factor of up to 100 have been observed.) Although there is 6 at.% solubility for H at room temperature within BCC niobium, the e-phase forms below 200K, which can distort the niobium lattice so much that the superconducting critical temperature is reduced to $T < 1.3$ K [1]. Q-degradation can be avoided by quenching through the dangerous temperature region of $75 \text{ K} < T < 150 \text{ K}$ thus eliminating the precipitation of the e-phase [2]. However, this is not a permanent "fix"; the only known permanent cure is degassing of the hydrogen from the niobium to low ppm levels.

Fig. 1 - Stress-strain plots of the elasto-plastic transition in high RRR Nb subjected to different heat treatments. The 800°C annealed sample shows a marked decrease in the apparent Young's modulus.

Fig. 1 - Courbes contrainte-déformation du Nb de RRR élevé après différents traitements thermiques. L'échantillon traité à 800°C montre une décroissance importante du module d'Young apparent.

The niobium cavities are degassed by heating in vacuum at a temperature above 500°C; at 800°C, for example, the hydrogen is degassed readily. However, it has recently been observed that these heat treatments substantially degrade the mechanical properties of the material. In addition to the obvious lowering of the yield strength with annealing, it was observed in some lots of material that the Young's modulus was also apparently reduced by a factor of 2 as a result of the hydrogen degassing at 800°C (Fig. 1). Intermediate anneals (e.g. 750°C or shorter time at 800°C) lead to intermediate behavior.

In an earlier report [3], it was shown that the Young's modulus determined from tensile test data is misleading in the case of this extremely soft material. Using the ultrasonic pulse-echo technique, and a reasonable assumption of isotropy (more later), the Young's modulus was determined to be typical for niobium, $E = 110 \pm 3$ GPa. The standard literature values range from $E = 105.4$ – 124.2 GPa [4]. Although the modulus was within the range of normal for all samples, there is an interesting trend of increasing modulus with annealing. The current study was undertaken to further investigate the changes in ultrasonic velocity with annealing. In particular, since single crystal niobium is anisotropic and changes in the crystallographic texture resulting from the rolling and annealing schedules may be responsible for the unusual behavior, careful texture analyses were performed.

Procedures

Material: Three different lots of niobium (denoted K, N and P) were obtained from Teledyne Wah Chang having nominally 99.95% purities (as determined by RRR measurements of 250 and 400 (lots K and P). Each series included as-received (rolled and recrystallized) material, as well as samples annealed for 6 hrs at 800, 750, & 700°C and 10 hrs at 600°C. The microstructures and crystallographic textures of the different samples were shown to be quite distinct.

Optical Metallography: Each of the samples were sectioned for examination transverse to the rolling direction in order to observe any banding or elongation of the microstructure due to processing. The samples were ground using SiC papers through 600 grit. Coarse polishing was performed using 6 μ m diamond slurry, followed by intermediate polishing using 1 μ m alumina slurry. The final polishing was performed using a procedure of repeated polishing and etching with 0.3 μ m alumina and/or 0.05 μ m colloidal silica and an etchant containing volumetric ratios 2:2:2:1 of distilled H₂O, HNO₃, HCl and HF.

Ultrasonic Velocity Measurement: A Panametrics 5072 Ultrasonic Pulser-Receiver was used in conjunction with a LeCroy 9420 Oscilloscope and 2.25 MHz transducers. Molasses was used as a couplant between the transducers and the samples for contact measurements. In addition, measurements of the compression wave speed were also made in a water immersion tank. Sample thicknesses were measured with a digital micrometer (to a relative uncertainty of $\sim 1/1500$) and ultrasonic velocities measured by determining the time-delay between the incident and echo waves reflected from the sample back-surface. The time delay was determined to a relative accuracy of 3×10^{-4} using the pulse overlap method, which amounts to finding the first peak in the correlation function. Velocities of the longitudinal (V_l) and shear (V_{s1} and V_{s2}) waves were used to determine the elastic constants C_{33} and C_{44} or C_{55} , respectively. The wave propagation direction was along the plate normal direction (denoted 3) in each case, while the shear polarization was along the rolling (1) or transverse (2) direction. For comparison purposes, a nominal density of 8.578 g/cm³ was assumed in all calculations. Comparing the values C_{44} and C_{55} , a partial assessment of the material's anisotropy may be made.

X-ray Diffraction Texture Analysis: A four-circle Scintag X-ray diffractometer and Cu-K α incident radiation (40 kV, 35 mA) was used to measure the crystallographic texture from the surface of the rolled niobium sheet samples. The (110), (200), (211) and (310) pole figures were collected on a 5° × 5° grid, up to a sample tilt of 80°. The texture data were analyzed using popLA (the preferred orientation package of Los Alamos) [5]. The raw pole figure data were corrected for defocusing using experimental data obtained from a randomly texture standard sample. Using a series of three pole figures from each sample, the complete orientation distribution (OD) was obtained using the WIMV subroutine of the popLA software. The presented full pole figures were recalculated from the OD. Subsequently, these textures were discretized for the modeling approach described below.

Electron Backscattered Diffraction (EBSD): EBSD measurements were performed in a JEOL 840 SEM, which has a conventional LaB₆ source and is equipped with an HKL Channel 5.0 EBSD system, including the Nordlys NL-1 high speed CCD camera. The main goal of the measurements was to determine if the texture within the bulk of the material was distinct from that of the surface and if this bulk texture evolves with annealing in a way that could explain the observed changes in ultrasonic velocity. The sample preparation for optical metallography (described above) was found to result in excellent electron diffraction patterns, although the etched surface is slightly rougher than is ideal for EBSD, since it leads to some shadowing of the patterns near grain boundaries. This was not a liability in the current context, where determinations of macrotexture were the primary goal. With this goal in mind,

rather coarse scans of the microstructure, 10 μm step size, were used to collect the required texture data. In order to make an orientation map of the microstructure, one region was scanned with a finer step-size of 2 μm .

Polycrystal Modeling: As with most metals (excepting W), niobium crystals are anisotropic. The anisotropy of cubic crystals is often given by the Zener parameter,

$$Z = \frac{2C_{44}}{C_{11} - C_{12}} \quad (1)$$

The single crystal stiffness coefficients for niobium given in Table I show that the Zener parameter of niobium is far from 1. In common with some other refractory metals (e.g. Mo, V) and distinct from many cubic metals (Cu, Ni, Fe, etc.) the Zener parameter is less than 1; thus, the $\langle 100 \rangle$ directions are the stiffest and the $\langle 111 \rangle$ directions are most compliant. In as much as a polycrystal is textured, the anisotropy of the single crystals will be manifest in the properties of the aggregate.

Table I - Single crystal elastic stiffness data for niobium (incl. Ta impurities) [6].

Tableau I - Coefficients d'élasticité d'un monocristal de niobium (contenant du Ta comme impureté).

	Nb	Nb 130 ppm Ta	Nb 1175 ppm Ta
C_{11} (GPa)	246.5	234.83	235.42
C_{12} (GPa)	134.5	120.68	121.52
C_{44} (GPa)	28.73	28.21	28.25
Z	0.513	0.494	0.496

Predicting the properties of the aggregate from the single crystal properties and the texture amounts to assuming some model for the distribution of stress (equilibrium) and strain (compatibility) between grains. As for many metals, the average of the upper (Voigt) and lower (Ruess) bounds (also known as the Hill average) is found to be a good approximation of the true response. The OD obtained from texture measurements is used as a weighting function for determining these averages.

$$\langle c \rangle_{\text{Hill}} = \frac{1}{2} (\langle c \rangle_{\text{Voigt}} + \langle s \rangle_{\text{Ruess}}^{-1}) \quad (2)$$

Results and Discussion

Microstructure

One finding that may prove to be important during future studies of these Nb sheets is the inhomogeneity of the grain size. There are bands of fine grains at approximately the quarter-plane of the sheets, which are apparent to the naked eye in nearly every sample. Figure 2 provides an example of a typical microstructure in this study where the banding is apparent in a single micrograph. Figure 3 shows the extensive grain growth that occurs upon annealing, including the appearance of some exceptionally large grains. Figure 4 graphically illustrates the sensitivity of the lineal intercept grain size to annealing temperature. In fact, the K sample annealed at 800°C obtained an average grain size approaching 350 μm . Given the microstructure heterogeneity in these samples (Fig. 2 and 3), it would not be surprising to find single grains with millimeter dimensions. Obviously, the metal's high purity contributes to its microstructure instability. Similarly, the batch-to-batch microstructure variation may be linked with details of lot purity.

Fig. 2 - A sample annealed 10 h at 600°C (lot P) showing typical microstructure heterogeneity, including the bands of smaller grains that could be seen with the naked eye on most of the samples examined. (The rolling direction is vertical.)

Fig. 2 - Echantillon traité 10 h à 600°C (lot P) montrant une hétérogénéité typique de microstructure, contenant des bandes de petits grains qui peuvent s'observer à l'œil nu. La direction de laminage est verticale.

Fig. 3 - An exceptionally large grain present in a sample (lot N) annealed 6h at the highest temperature, 800°C.

Fig. 3 - Un très gros grain présent dans un échantillon du lot N, traité 6h à 800°C.

Fig. 4 - Lineal intercept grain size as a function of annealing temperature for the three lots of high-purity niobium that were examined during this study.

Fig. 4 - Taille de grains déterminée par la méthode de l'intercept linéaire, pour les trois lots de niobium de haute pureté examinés dans la présente étude.

Ultrasonic Velocities

The longitudinal velocities of the samples are relatively insensitive to annealing (see Fig. 5). There may be a very subtle decrease in the longitudinal velocities of some lots, and though the experimental accuracy seems adequate, sample-to-sample variations preclude a conclusive statement on this point. In the case of the shear wave speeds, there is a clearer trend in the data. For each of the sample sets, the velocity of the shear wave polarized in the rolling direction remains essentially constant, while the wave polarized in the transverse direction trends upwards (Fig. 6). In one of the sample lots, measurements were performed for shear waves polarized in the 45° orientation and the intermediate values obtained from these measurements also

Fig. 5 - Ultrasonic velocity measurements for the longitudinal waves in lots K, N and P show the longitudinal velocity to be essentially constant with annealing for each lot.

Fig. 5 - Mesures de vitesse ultrasonore pour les ondes longitudinales dans les lots K, N et P, montrant que la vitesse longitudinale est indépendante des conditions de recuit pour chaque lot.

tend towards higher velocity with annealing (Fig. 6). Essentially, there is an increase in the birefringence (or anisotropy of the shear wave velocities) with annealing for each of the sample lots. As such, texture evolution appears a likely explanation, however, preliminary texture measurements had shown the texture to remain essentially constant with annealing [3].

Crystallographic Texture

The pole figures obtained from X-ray diffraction measurements have peak intensities of about 4 times random indicating a moderately strong texture resulting from the cold-rolling and recrystallization processes (e.g. Fig. 7). The texture strength (as indicated by the mean squared intensity value in the OD) was

Fig. 6 - Ultrasonic velocity measurements for the shear wave polarized in the sheet's rolling (RD) and transverse (TD) directions for the sample series K, N and P.

Fig. 6 - Mesures de vitesse ultrasonore pour les ondes de cisaillement polarisées selon la direction de laminage de la plaque (RD) et la direction transverse (TD) pour les échantillons des lots K, N et P.

Fig. 7 - (110) and (200) pole figures collected using X-ray diffraction of the sheet surfaces of the as-received and annealed material (lot N) demonstrate that the surface texture does not evolve with annealing. Contours represent multiples of the random distribution intensities.

Fig. 7 - Figures de pôle (110) et (200) obtenues aux rayons X sur la surface des plaques dans les états de réception et après recuit (lot N), montrant que la texture superficielle ne change pas avec le recuit. Les contours représentent des multiples des intensités de la distribution isotrope.

Fig. 8 - Pole figures from high-purity niobium batches K, N, and P annealed 10h at 600°C (the selected degassing treatment) show significant distinctions in the textures that are most likely due to variations in the cross-rolling procedure during production.

Fig. 8 - Figures de pôle du niobium de haute pureté (lots K, N et P) recuits 10h à 600°C, montrant les modifications de textures vraisemblablement produites par le procédé de production par laminage croisé.

between 2 and 3 for all of the measured samples. Analysis of the full orientation distribution (not shown for brevity) reveals that there are two main components of the texture: $\{111\}$ || sheet plane (also known as the γ -fiber in the literature concerned with BCC rolling textures [7]), and the $\{001\}<100>$ cube texture component. The former is a typical BCC rolling texture component, while the latter seems unusual to the present authors. There is little discernable change in the surface texture with annealing (Fig. 7). Although there is little change in texture with annealing, the current investigation highlights the fact that the texture does vary from one batch to another (Fig. 8). It is suspected that the cross-rolling procedure employed by the manufacturer is not held constant from one batch to another.

The EBSD measurements collected from the sheet cross-sections reveal a bulk texture similar to that of the surface measurements, with a notable distinction that the $\{100\}<001>$ component is weaker relative to the γ -fiber (Fig. 9). This same observation was made by another group studying e-beam welds of high RRR Nb sheets [8]. Also in contrast with the measurements of the sheet surface texture using X-ray diffraction, it was found that the texture within the bulk of the material evolves with annealing, such that the texture in the samples annealed at 800°C is significantly stronger than that of the as-received material (see Fig. 9b). Thus, it is consistent with the increasing anisotropy that was observed by ultrasonic velocity measurements. Orientation imaging map obtained using EBSD (Fig. 10) suggests the possibility that the banded microstructure is due to some orientation effect.

Polycrystal Modeling

As described above, if the single crystal properties and the crystallographic texture are known, the elastic behavior of the polycrystal may be predicted on the basis of simple averaging schemes (Eq. 2). From the predicted elastic stiffness components, we can also predict the ultrasonic velocities. If an idealized random texture is used to predict the elastic coefficients of isotropic niobium (Table II), it can be seen that the measured velocities of the current study are in the same range as the isotropic values predicted by the Hill model. However, there are measurable distinctions between the samples that may be connected to the evolution of texture.

In Table III, are shown the simulated moduli and ultrasonic velocities on the basis of the Hill approximation and the textures, which were obtained using EBSD. Given the symmetry of the textures, it is not surprising that the predictions of the elastic moduli exhibit orthotropic symmetry as well. Only the C_{33} , C_{44} and C_{55} coefficients are reported here, since they are the ones for which experimental data is available for validation.

As can be seen in Table III, the shear velocities in the as-received conditions are close to one another for both the K and P samples, while there is an increased birefringence in the annealed samples, due to the increase in texture. Thus incorporation of the bulk texture is important to fully understand the properties of these materials. If the texture determined at the surface is used for the predictions, we observe no change with annealing. Furthermore, the predicted anisotropies are not observed experimentally (Table IV). For one thing, it has already been mentioned that the surface texture contains a much more significant volume fraction of the cube component than is present within the bulk (see Figs. 7-9).

Fig. 9 - Crystallographic texture measurements from the sheet cross-section made by EBSD, from the high-purity niobium lot K (a) as-received and (b) annealed 6 h at 800°C, showing the increase in texture strength that is not apparent from the X-ray surface measurements. (Contours are multiples of a random distribution in levels of 0.5.)

Fig. 9 - Texture cristallographique par EBSD sur la section des plaques pour le niobium de haute pureté du lot K (a) état de réception et (b) recuit 6 h à 800°C, montrant l'augmentation de l'intensité de la texture non visible aux rayons X sur la surface. Les contours sont des multiples des intensités de la distribution isotrope par niveaux de 0.5.

Fig. 10 - OIM map of as-received P sample shows bands of fine and coarse grains. The different colors (shades of grey) represent orientations as shown in the legend, where the Euler angles are according to Bunge's convention (ϕ_1 , Θ , ϕ_2). The white spots represent positions where surface roughness obscured the EBSD detector.

Fig. 10 - Carte d'orientation de l'échantillon P dans l'état de réception montrant des gros et des petits grains. Les différents niveaux de gris représentent les différentes orientations. Les taches blanches sont des zones où la rugosité de surface empêche la détection par EBSD.

Table II - Predicted isotropic stiffness coefficients and ultrasonic velocities of high purity niobium, assuming a random texture.

Tableau II - Coefficients de rigidité isotropes et vitesses ultrasonores dans du niobium de haute pureté, calculés en supposant une texture isotrope.

	Voigt	Ruess	Hill
C_{11} (GPa)	224.5	219.4	221.9
C_{12} (GPa)	145.5	148.1	146.8
C_{44} (GPa)	39.7	35.7	37.7
V_l (m/s)	5116	5058	5086
V_s (m/s)	2151	2040	2097

Table III - Predicted stiffness coefficients and ultrasonic velocities on the basis of EBSD textures.*Tableau III - Coefficients de rigidité et vitesses ultrasonores calculées à partir des textures obtenues par EBSD.*

	N-series	600°C, 10hr K	800°C, 6hrs P
C_{33} (GPa)	230.8 ± 0.5	226.3	231.9
C_{44} (GPa)	33.4 ± 0.2	35.2	32.8
C_{55} (GPa)	34.1 ± 0.2	36.1	32.8
V_l (m/s)	5187	5136	5199
V_{s2} (m/s)	1973	2026	1955
V_{s1} (m/s)	1993	2051	1955

Table IV - Predicted stiffness and velocity data for N-series (average) and selected P and K samples, based upon surface texture measurements.*Tableau IV - Rigidité et vitesse calculées pour la série N (moyenne) et des échantillons de P et K, à partir des mesures de texture superficielle.*

	as-received K	800°C, 6hrs K	as-received P	800°C, 6hrs P
C_{33} (GPa)	216.6	219.0	220.0	219.0
C_{44} (GPa)	40.8	40.4	38.9	40.4
C_{55} (GPa)	40.3	38.0	38.4	38.0
V_l (m/s)	5025	5053	5064	5053
V_{s2} (m/s)	2181	2170	2130	2170
V_{s1} (m/s)	2168	2105	2116	2105

There are still unanswered questions regarding the significant increase in the birefringence that are not quantitatively modeled, however, the general trends have been captured. In short, this study has reemphasized [9] the fact that ultrasonic velocity measurements are a very sensitive way to detect changes in texture. Furthermore, given the high atomic number of niobium and, therefore, shallow penetration depth of X-rays, the changes in texture that are detected by ultrasound would not be possible from normal sheet surface texture measurements using Cu K_α X-ray radiation most commonly available in X-ray diffraction laboratories. It remains to be seen if a more penetrating radiation would provide greater insight, however, the most obvious choice, Mo K_α would likely have problems with high background due to x-ray fluorescence.

It is mentioned above that the textures primarily consist of the γ -fiber along with a minor contribution of the cube component, so the impact of these ideal texture components is included for comparison (Table V). It is shown that simply accounting for

Table V - Predicted stiffness and velocity data on the basis of ideal texture components for comparison.*Tableau V - Rigidité et vitesse calculées sur la base des composantes idéales de texture pour comparaison.*

	Ideal γ -fiber	Ideal cube	0.8 γ + 0.2 cube
C_{33} (GPa)	2.101	2.465	2.158
C_{44} (GPa)	0.477	0.287	0.410
C_{55} (GPa)	0.477	0.287	0.41
V_l (m/s)	4949	5360	5016
V_{s2} (m/s)	2283	1820	2186
V_{s1} (m/s)	2283	1820	2186

these texture components can give values quite close to those measured in this study (0.8 γ -fiber + 0.2 cube component). Note that other minor texture components must be responsible for the birefringence, since these ideal components all exhibit $C_{44} = C_{55}$.

In the final analysis, there are only subtle changes in the velocities, and even the shear wave velocities only change by about 1% on average. Furthermore, if there is a strong texture, we will observe some velocities to be increased, while others are decreased (Table V). Obviously, there are batch-to-batch variations that cannot be explained on the basis of texture. For example, lot K exhibits longitudinal and shear velocities, which are greater than the other two sample lots.

It is suggested that impurity effects may be responsible for the variation between lots. Ta is a known impurity in these Nb sources and, as shown in Table I, there is documented evidence that Ta can significantly affect the moduli of Nb, even in the ppm range [6]. The trend would suggest that the K samples have less Ta than the N and P lots. Likewise, H additions can have an influence, although it is small (~1% change per 1000 ppm) [10] and we would expect a regular trend to be observed between the various lots as the H levels are decreased due to out-gassing treatments at the annealing temperatures studied. Further evidence that there may be differences in the impurity levels is provided by the fact that the grain size of the K lot is significantly larger than the grain size of the lots N and P (Fig. 6). Impurities are known to have a significant effect in stagnating grain growth [11]. Since the grain sizes of lots N and P obtain the level at which the K lot begins, we can say that the grain size itself is not responsible for the observed effect.

Summary

The effect of annealing on the ultrasonic velocities and crystallographic texture of three lots of high RRR niobium plate material has been evaluated. The velocity measurements reveal that the elastic properties of the material are approximately constant with annealing and within the range of values published in the literature. Thus, the unusually low *apparent* Young's modulus, formerly reported in the same materials, was shown to be an artifact of the tensile testing technique due, in fact, to the low yield strength in some of the annealed samples.

In detail, there is a trend of increasing velocity with annealing, for shear waves propagating through the sheet thickness and polarized in the sheet transverse direction. Preliminary examination of the crystallographic texture via X-ray diffraction of

the sheet surface showed the texture to be essentially unchanged with annealing. However, careful analyses of the crystallographic texture using SEM-based electron backscattered diffraction (EBSD) have revealed a subtle, but systematic change in the texture. On the basis of Hill's averaging scheme, this texture change can explain the trend observed in the velocities. Thus, measurements of ultrasonic velocity represent a non-destructive evaluation tool, which is extremely sensitive to subtle changes in the texture of high RRR niobium that would otherwise require sectioning of the sheet to detect.

There are variations in the elastic properties from one lot to another, and these are currently attributed to the effects of impurities, such as Ta and H. □

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Acknowledgments

This work was supported by a grant from Reference Metals Company to the University of Virginia and the U.S. Department of Energy under Contract No. DE-AC05-84ER40150. We would also like to thank Mr. YUN Jo Ro, for his invaluable help discussing and tutoring us in the use of the EBSD equipment and data analysis.

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