

Estimative of the stacking fault energy for a FeNi(50/50) alloy and a 316L stainless steel

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Abstract: The effect of high energy milling on powders of a FeNi (50/50) alloy and a 316L stainless steel has been evaluated by means of X-Ray Diffraction (XRD). The average microstrain as function of the milling time (1/2h, 1h and 8h) was determined from XRD data. The displacement and broadening of the (XRD) peaks were used for estimate the stacking fault energy (SFE), using the method of Reed and Schramm. It was estimated SFE=79 mJ/m² for the FeNi (50/50) alloy and SFE=14 mJ/m² for the 316L stainless steel. The better experimental conditions for determining the SFE by XRD are discussed.

Introduction

The Stacking Fault Energy (SFE) is a very important parameter for FCC (face centered cubic) metals and alloys. Higher the SFE, higher is the tendency to occur cross-slip of dislocations, and this dictates the behavior of FCC metals when submitted to plastic deformation (caused by, for example, milling or rolling). The most accurate methods for SFE determination require TEM (Transmission Electron Microscope). The TEM related methods are “direct”, i.e., the Stacking Fault Energy is estimated from the direct observation of the distance between dissociated partial dislocations [1,2] or from dislocation nodes after plastic deformation [3].

In the present day, the only SFE determinations considered as reliable are by means of weak beam method (TEM) [1,2] or high resolution electron microscopy (HREM) [4]. However, these methods are laborious, expensive and not readily accessible.

One of the objectives of the present study is to show the possibility of SFE determination just using peak position and FWHM (full width at half maximum) of the X-Ray Diffraction (XRD) peaks of deformed (or cold worked) and well annealed FCC metals. An obvious additional advantage of the XRD method is its simplicity.

The big disadvantage of the XRD method is to be an indirect method: a “calibration” with reliable SFE values, used as reference, is necessary. This problem is overcome since SFE reference values are chosen from determinations using weak beam method [2,5]. The typical metals used as reference for calibration are Ag, Au, Cu and Al [6-9]. In the past, Reed and Schramm [6-8] already successfully employed the XRD method, but they unfortunately used as reference overestimated SFE values for Ag, Au, Cu and Al, and this maybe contributed for some discredit of the SFE XRD method.

In the present study the XRD SFE method will be employed for a NiFe (50/50) alloy (reportedly having “high” SFE, ~90 mJ/m² [8]) and a Stainless Steel 316L (reportedly having

“low” SFE, 17 mJ/m² [9]). Both alloys have very similar lattice parameters, 3.60 Å, and Fe and Ni as main alloying elements.

Experimental

Both materials FeNi (50/50) and Stainless Steel 316L were processed and examined in the same way. The starting materials were commercial pre-alloyed powders. Those powders were milled in a high energy mill, during ½ hour, 1 hour and 8 hours. Reference samples (the well annealed materials) were obtained by sintering. The powders were pressed with 600 MPa and pressed samples were sintered inside a dilatometer. Samples were heated (20K/min.) up to 1300°C under dynamic argon. The sintering time at this temperature was 60 minutes. XRD Spectra were obtained for the peaks (111) and (200), using CuK α radiation (step=0.01 degrees, and 3s of acquisition time per step) in a Rigaku Diffractometer model Multiflex with monochromator of graphite. These XRD spectra were examined using the method described by Warren [10] for determination of SFE probability, based on peak displacement after cold working.

Theory: Description of the XRD SFE method

The stacking fault probability (α) of a FCC crystal can be found directly from the change of position of the X-Ray Diffraction peaks due the plastic deformation, using the expressions (1) and (2) [7,8,10], where 2θ is given in degrees:

$$\Delta 2\theta^{\circ} = (2\theta_{200} - 2\theta_{111})_{def} - (2\theta_{200} - 2\theta_{111})_{ann} \quad (1)$$

$$\Delta 2\theta^{\circ} = \alpha \cdot \frac{-45\sqrt{3}}{\pi^2} \left(\tan \theta_{200} + \frac{1}{2} \tan \theta_{111} \right) \quad (2)$$

The stacking fault energy (γ) is given by Eq. (3) [6-8]:

$$\gamma = P \cdot \frac{a \cdot G_{111}}{\pi\sqrt{3}} \cdot \frac{\langle \varepsilon^2 \rangle_{111}}{\alpha} \quad (3)$$

where a is the lattice parameter, G_{111} is the shear modulus for the (111) plane, and P is a non-dimensional “proportionally constant”, experimentally found to be in the range (4.6 to 6.6) [6-9]. The proportionality constant should be described in more detail: P is $K_{111} \omega_0$ [6], where ω_0 is the width of the separation of partial dislocations for an intrinsic stacking fault and K_{111} depends on crystal geometry. The method of Reed and Schramm [6-8] assumes that the product $K_{111} \omega_0$ is a constant, and this seems to be valid for Ag, Cu, Au and Al. Thus, the constant P should be estimated from the most reliable literature data concerning the determination of stacking fault energy. Selected values are respectively (mJ/m²): Ag (16) Au (32) Cu (41) Al (150) [2,4,5].

An example of determination of the Proportionality factor P is shown in Figure 1. The data of Borges et al [9] was used as basis for Figure 1. The regression coefficient (i.e., the R-value) obtained adjusting for a straight line the data of Figure 1 is $R=0.99927$. The slope of the straight line (Figure 1) is $(\sqrt{2}/10\pi) K_{111} \omega_0$. This new determination of P changes the $P=4.6$ found by Borges et al [9] to $P=5.0$.

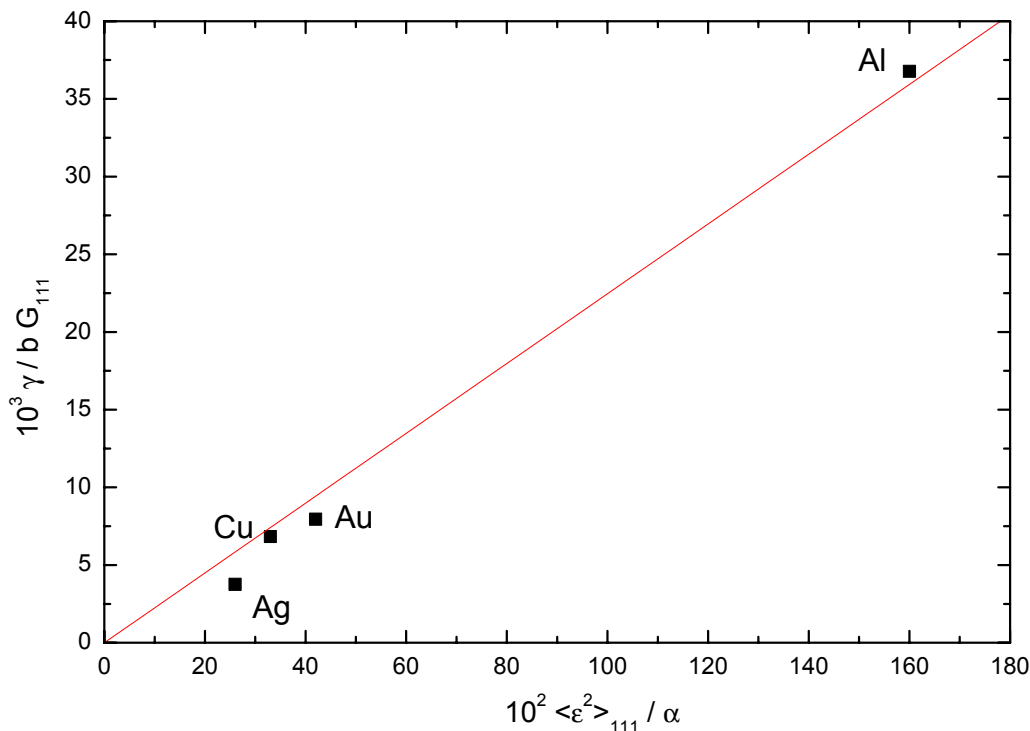


Figure 1. Determination of the Proportionality factor P, using data from Borges et al [9], but selected values for SFE, as discussed in the text. b is the Burgers vector ($b = a / \sqrt{6}$). It is found $P=5.0$ from the slope of this curve.

The mean square strain $\langle \epsilon^2 \rangle_{111}$ is estimated from the peak broadening of the diffraction line for the peak (111), as seen in Eq. (4).

$$\langle \epsilon^2 \rangle_{111} = \frac{(\Delta d / d)_{111}^2}{1 + 2\nu^2} \quad (4)$$

where d is interplanar distance, ν is the Poisson coefficient. $\Delta d/d$ is found by differentiating the Bragg law ($n \lambda = 2 d \sin \theta$), giving $\partial d \cdot \sin \theta = -d \cdot \partial \theta \cdot \cos \theta$, see Eq. (5).

$$\left(\frac{\Delta d}{d} \right)^2 = B^2 \cdot \text{ctg}^2 \theta \quad (5)$$

where B is the FWHM, θ is the Bragg angle for diffraction peak with index hkl , and ctg is the cotangent. The elastic energy stored during the deformation W is related to the x-ray peak broadening (Eq. 6). E_{hkl} is the Young modulus for the direction perpendicular to the plane (hkl).

$$W = \frac{3}{2} \cdot E_{hkl} \cdot \frac{(\Delta d / d)_{hkl}^2}{1 + 2\nu^2} = \frac{3}{2} \cdot E_{hkl} \cdot \langle \epsilon^2 \rangle_{hkl} \quad (6)$$

According to Burgahn et al [11,12], the residual microstresses, $\sigma_{\text{micro}}^{\text{RS}}$ also can be estimated, with equation (7):

$$\sigma_{micro}^{RS} = E_{hkl} \cdot \langle \varepsilon^2 \rangle_{hkl}^{1/2} \quad (7)$$

The shear modulus for the stainless steel is $G_{111} = 0.65 \times 10^{11} \text{ Nm}^2$ [7] and for the FeNi(50/50), $G_{111} = 0.50 \times 10^{11} \text{ Nm}^2$ [8,13]. It was assumed here $\nu=0.3$, a typical value for stainless steels.

Results and Discussion

It was determined that the lattice parameters of both alloys, before deformation, are 3.60 Å. We confirmed the problem reported by Reed and Schramm [8], concerning the reduction of lattice parameter in NiFe alloys due to plastic deformation. This leads to error in measurement [8] and, to avoid this, the SFE was determined after short time of milling. Besides, other phenomena (for example, mechanical alloying) may occur if the samples (powder) are overmilled. It was noted, for the FeNi 50/50 alloy, that the (111) peak presented asymmetry after ½ h of milling. After 1 hour, this asymmetry was not observed. Thus, the analysis of the experimental XRD data indicated that the samples milled during 1 hour should provide a better estimate.

It was found that Lorentzian distribution allows a better fitting for the well annealed samples, whereas Gaussian distribution may allow a little better fitting for the deformed samples. After some comparison, it appeared that Lorentzian provided the better adjust. Nevertheless, both methods – Lorentzian and Gaussian – pointed out the same center of peak. It was found to be essential that the center of peak be determined using peak fit (Lorentzian or Gaussian), because the SFE estimated values are quite sensitive to peak position; and small inaccuracy on peak position may lead to big discrepancies on the measured SFE value. The size of $K\alpha_2$ is half of $K\alpha_1$ thus, in principle, all FWHM is due to $K\alpha_1$ radiation. However, the background should be very small to assure that $K\alpha_2$ is not contributing for the FWHM and this only can be achieved if the time of data acquisition is sufficiently high.

Using the microstrain of Table I and Eq. (1-3), the SFE was estimated (the data of the samples submitted to 1hour of milling were used): For FeNi 50/50, $\text{SFE}=79 \text{ mJ/m}^2$, compatible with the literature values [8] and confirming that FeNi alloys have high SFE. By another hand, it was obtained $\text{SFE}=14 \text{ mJ/m}^2$ for the stainless steel, in very good agreement with the values reported by Borges et al (17 mJ/m^2) [9], and by Kestenbach (20 mJ/m^2) [14] confirming the 316L stainless steel as a low SFE material.

If Fourier analysis would have been employed to determine the microstrain, instead of FWHM, this would not improve very much the SFE estimate, because the obtained values depend fundamentally on peak position, as it was observed in the present study. Nevertheless, it is conceivable that Fourier analysis could lead to better estimates of the microstrain, because all information from the “tail” of the peak would be used, and this is an interesting subject to further studies: a comparison between microstrain values obtained with FWHM and Fourier analysis.

Table I. Microstrain ($10^3 \langle \varepsilon \rangle^{1/2}$) for the FeNi 50/50 and Stainless Steel 316 L, as function of the milling time, for the planes (111) and (200).

Time	FeNi 50/50 (111)	FeNi 50/50 (200)	316L (111)	316L (200)
½ h	2.3	3.0	2.8	1.0
1 h	3.5	5.6	3.0	3.2
8 h	3.6	6.2	3.1	4.1

The values of Table I show that the microstrain increases with milling time, and that the amount of microstrain as function of milling (or deformation) is different for each analyzed plane, but that similar trend is observed for both, high and low SFE materials.

The stored energy in the deformation and residual microstresses can be estimated with Eq. (6) and (7) and the data of Table 1, since E_{hkl} values are available, but E_{hkl} data about these specific alloys are scarce in the literature. A rough estimation of the dislocation density using the stored energy in the deformation is also possible [15], since several assumptions about the dislocation arrangement are employed.

Conclusions

The proportionality factor P was redetermined, using more recent and reliable data. It was found $P=5.0$, close to the $P=4.6$ value of Borges et al [9], and confirming that the $P=6.6$ of Reed and Schramm [6-8] was overestimated. As consequence, all SFE values reported by Schramm and Reed [6-8] should be reduced to ~75% of the reported value.

It was found that the SFE found by XRD is function of the time of milling. Overmilled powders lead to incorrect values. By another hand, if the powders are milled for insufficient time this results in underestimation of peak displacement and, as consequence, the SFE values result overestimated.

The SFE values - 79 mJ/m^2 for FeNi 50/50 and 14 mJ/m^2 for 316L stainless steel – confirm these materials as “high” (FeNi 50/50) and “low” (316L stainless steel) SFE.

The described XRD method is quite simple, rapid and cheap, and can easily be employed for estimation of SFE, since reliable reference SFE values are used. The characteristics of the SFE XRD method make it to be particularly suitable for systematic studies where several alloys with different chemical composition are compared.

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