Intensity Correction and Pole Figure Measurement of Copper Metallic by XRD

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Abstract: In this research work the process of data intensity correction and pole figure measurement were investigated and analyzed by using X-Ray Diffraction (XRD) technique. Different corrections were studied, to see their influence on the crystallographic texture analysis. Specific software, which is Labotex was used to determine the pole figure for copper (Cu) metallic after which the data corrections have been made. The copper, Cu sample was simply prepared by a low cost method Mini Mill 2 Panalytical and the sample was rotated for 10 minutes by grinding the sample at high speed of 250 rpm.

Keywords: X-ray diffraction, intensity correction, pole figure.

1. INTRODUCTION

Texture measured of the pole figures is to give some information on the Cu sample microstructure, the arrangement of grain orientation of the copper metallic. Before the pole figure can be evaluated, several correction to be applied such as defocusing, background and absorption correction [1, 2]. Then the source of X-ray and the detector are oriented so that a given value of 2θ is specified. The stage of the cradle is tilted and rotated symmetrically to determine orientation distribution of crystalline grain in a polycrystalline material [3]. To perform the correction of defocusing, the best procedure involves measuring the intensity from a reference sample with random texture composed of the same material of the studied sample. However, the intensity drop also depends on the alignment of sample and goniometer as well as the size of the collimator, receiving slit, the tilting angle ψ of a sample and the Bragg angle θ of diffraction. The diffraction intensity decreases with the increase of tilting angle ψ, caused by defocusing. The pole figures must be corrected have background intensity. The background intensity to measure on both sides for each peak copper sample [4].

2. EXPERIMENTAL TECHNIQUE

The copper, Cu sample was prepared by Mini Mill 2 Panalytical and the sample was rotated for 10 minutes by grinding the sample at high speed of 250 rpm. Herzog press Panalytical makes pressing application where the hydraulic pump is operated for a pressing to 10 tons. The crystalline grain size (D) of the sample was determined by using Scherrer formula.

$$D = \frac{0.9\lambda}{\beta \cos\theta} \quad (1)$$

Where, λ is wave length of X-ray = 1.5406(Å), β is FWHM (full width at half maximum), θ is the degree of the diffraction peak and D is crystallite size. In this study, polycrystalline copper metallicis an important material with face centre cubic crystal structure. The average grain size of the sample was approximately 35.5 (nm). XRD of model Panalytical Empyrean is used to determine peak position that operating at 45 kV and 45 mA, with a Cu X-ray tube (λ = 0.15406 nm) [5]. The incident beam is collimated with a 1 mm. The detector set-up is defined by four receiving slits 4 mm of acceptance and a proportional detector. In the present diffraction pattern of XRD, three dominant peaks of 2θ is approximately equal to 43.85˚, 50.82˚ and 74.65˚ which is corresponding to the (111), (200) and (220) planes of Copper metallic as shown in Figure **1** of cubic crystal structure.

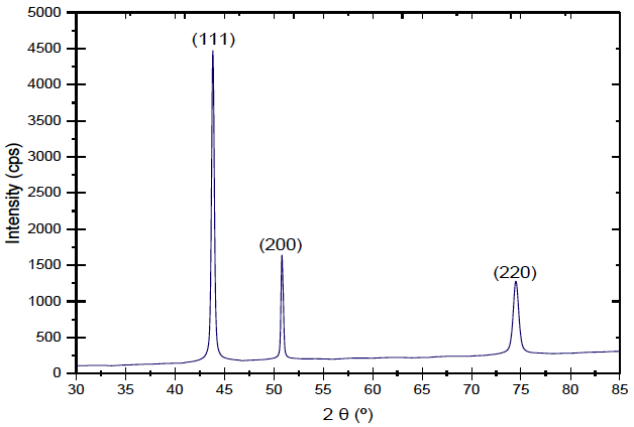

Figure 1: X-ray diffraction patterns of copper metallic sample.

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3. INTENSITY CORRECTION

Before performing quantitative texture measurement, different corrections have to be performed on the raw data, namely defocusing, background and absorption correction as shown in Figures **2** and **3**. The correction factor at each ψ angle depends on the 2θ position and width or size of the receiving slit and collimator because the smaller the collimator size and larger the receiving slit, the smaller is the defocusing error, though larger receiving slits decrease the angular resolution. Defocusing correction required to increase the intensity towards the edge of the pole figure [6, 7]. Intensity lost from detector must be compensated from using the defocusing correction. The defocusing correction depends on the Bragg angle θ, correction curves of other materials, Copper powder for correction Cu sample can be generally corrected with the correction curves derived from a Cu powder [7, 8]. Absorption correction when a copper sample analysed in transmission geometry is chi (ψ), the path length of X-ray within the copper sample increase much more than the decrease in diffracted intensity, independent on the Bragg angle [9, 10]. The influence of absorption on the peak exactly ψ = 0º to 30º because it came up with the correction as shown in Figure **3**. The intensity decreases with increasing chi ψ, it must be measure the background (Bg) correction at either side of the Bragg peak, e.g. 2θ = 43.80˚ of the peak Cu (111) at background position select Bg_1 (2θ= 40˚) Bg_2 (2θ = 46˚) as shown in Figure **2**. The first peak of copper is very strong intensity; it means that the intensity decreased versus 2θ must be corrected by the background correction for each peak because the intensity interaction with air and electronic noise as shown in Figure **3**.

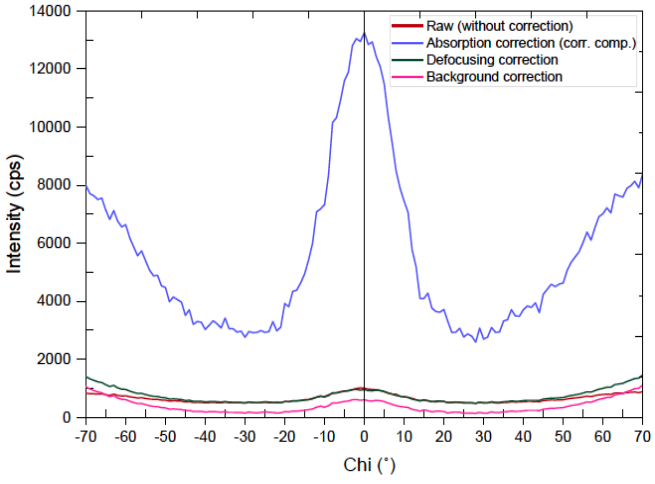

Figure 2: Three corrections of the intensity copper metallic (111).

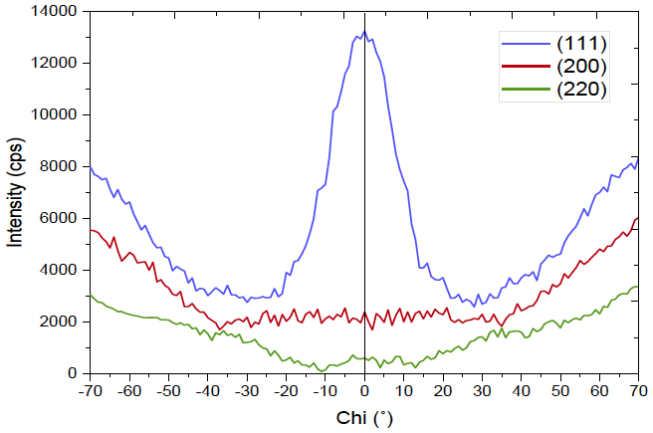

Figure 3: Three corrections or (perfectly correction) for the intensity of three planes Cu (111), (200) and (220).

4. POLE FIGURE MEASUREMENT BY XRD

After several data correction of the intensity is available for determined the pole figure (PF). The most important data file is the experiment pole figure file that needs to be imported and, if necessary converted from other conventions. Pole figure is scanned by measuring at a fixed 2θ position angle (crystal orientation), a complete ϕ scan (spins the sample about its normal) can be performed at each specific tilt angle (ψ).Pole figure measurement is considered as an important process needs to be undertaken to get a successful characterization of the sample [9]. The latter represents the orientation distribution of one plane of the crystal lattice. Pole figures were performed Bragg peak to determine the true value of 2θ as shown in Figure **1**. Check for different value of ψ (from -70° to 70°) the angle between the normal to the sample. It took the value ψ (from 0˚ to 70˚) and the normal to the diffracting planes also reflection the pole figures are most accurate in the centre ψ = 0º. The value ϕ (from 0˚ to 90˚) for Cu (face-centre cubic) Moreover, for a given angle ψ, the X-ray intensity is independent of the angle ϕ because of the rotational symmetry a round the S_3 axis as shown in Figure **4**.

5. CONCLUSIONS

This paper has explained the data plotting and orientation analysis of pole figure data and data correction processing of intensity such as defocusing and background and absorption correction. The orientation texture can be analysed from the pole figure data obtained by pole figure measurement. The maximum and minimum intensities for Cu (111) were corresponding to ψ= 0˚ and 70˚, respectively. In addition, the maximum and minimum intensities for

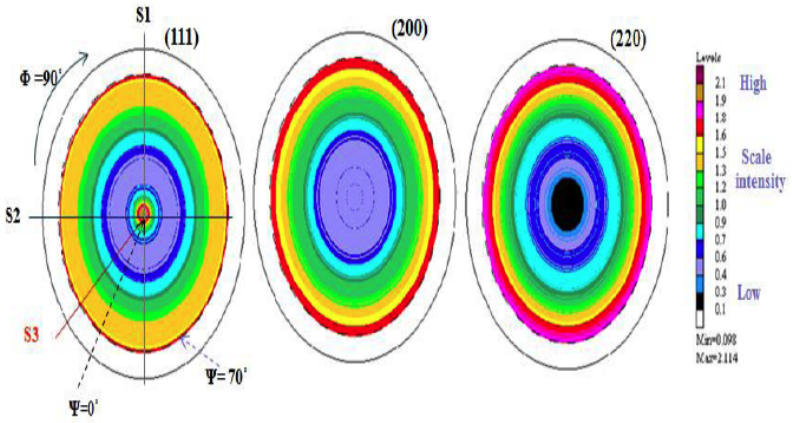

Figure 4: Shows the Pole figure of Cu metallic (111), (200) and (220) after three correction, ψ = 70°.The S_3 axis is always along the direction of the specimen normal, and S_1 and S_2 in the loading and transversal direction respectively.

both planes Cu (200) and (220) were found to be ψ= 70° and 0°, respectively.

The texture analysis is influenced by the correction factor at each ψ angle, which is dependent on the 2θ position and width or size of the receiving slit and collimator.

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