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TEXTURE MEASUREMENT BY NEUTRON DIFFRACTION FOR A1 NONSTANDARD STEEL BARS

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ABSTRACT

TEXTURE MEASUREMENT BY NEUTRON DIFFRACTION FOR A1 NONSTANDARD STEEL BARS. Texture measurements are used to determine the orientation distribution of crystalline grains in a polycrystalline sample. A material is termed textured if the grains are aligned in a preferred orientation along certain lattice planes. The texture is usually introduced in the fabrication process (e.g. rolling of thin sheet metal, deposition, etc.) and affects the material properties by introducing structural anisotropy. The pole figure is the starting point of neutron texture analysis. Texture properties in an non-standard austenite Fe-Ni low carbon alloy were measured by the neutron diffraction method with the PD/Residual Stress Diffractometer DN3 apparatus in BATAN. In this paper, effects of hot rolling on the texture are studied using ODF plots generated by MAUD application code developed by Lutteroti. It was found that The ODF plots for the A1 as cast and 1200 °C hot rolled specimens show typical patterns for recrystallized and rolled cubic structure. The pole figures for this plate are consistent with Face Centered Cubic (FCC) rolling textures

Key words : Neutron, Pole figures, ODF, Low carbon alloy, Fe-Ni based alloy

ABSTRAK

PENGUKURAN TEKSTUR DENGAN *NEUTRON DIFFRACTION* UNTUK BAJA NON STANDAR A1. Pengukuran tekstur digunakan untuk menentukan distribusi orientasi butir-butir kristal dalam sebuah sampel polikristalin. Suatu benda atau bahan disebut sebagai memiliki tekstur jika arah butiran kristal dalam bahan terorientasi atau selaras dengan suatu arah orientasi yang diutamakan (*preferred*) di sepanjang bidang kisi tertentu. Tekstur biasanya terjadi atau terbentuk pada saat proses fabrikasi bahan (misalnya pada pengerolan lembaran logam tipis dan deposisi) dan akan mempengaruhi sifat bahan tersebut karena mengakibatkan anisotropi struktural pada sifat sifat fisis bahan. Langkah awal analisis tekstur menggunakan metode neutron disajikan melalui suatu *plot* yang diistilahkan sebagai *pole figure*. Sifat tekstur pada paduan non standar austenit Fe-Ni karbon rendah diukur dengan metode difraksi neutron menggunakan instrumen *DN3 PD/ Difractometer Residual Stress* di BATAN. Dalam tulisan ini, efek hasil pengerolan panas pada tekstur spesimen baja non standar A1 telah dianalisis dengan menggunakan *plot ODF* yang dihasilkan oleh kode aplikasi MAUD yang dikembangkan oleh *Lutteroti Maud*. Hasil analisis menunjukkan bahwa *plot ODF* untuk spesimen A1 *ascast* dan spesimen A1 hasil pengerolan panas pada suhu 1200 °C merupakan suatu pola yang khas untuk spesimen berstruktur kubik yang telah mengalami rekristalisasi dan pengerolan. Disimpulkan bahwa pola tekstur untuk pelat baja ini ternyata konsisten dengan pola tekstur untuk suatu kristal yang memiliki simetri kristal kubik berpusat muka (*FCC*).

Kata kunci : Neutron, *Pole figures, ODF*, Paduan karbon rendah, Paduan berbasis Fe-Ni

INTRODUCTION

Fe-Ni- based Low Carbon steel (LCS) are still the subject of many research activities. Fe-Ni- based Low Carbon steels exhibit outstanding resistance against segregation [1]. The segregation only occurs under extensive application of external forces and with the help of a fast plastic deformation [2]. Hot rolling is one of the method utilized to obtain steels in a desired size. However when Fe(Ni- LCS) samples are hot rolled at 1200 °C and achieved 40% reduction, both the initial fabrication of the LCS (as cast) and the subsequent hot rolling process introduced residual stress in the samples, as well textures are formed in the materials.

Texture is an important structural parameter in all polycrystalline materials [3]. Traditionally texture is used to determine to anisotropy of properties of many important engineering materials. Polycrystalline materials

contain many millions of grains. Each grain in a specimen has a different crystallographic orientation from its neighbours. Materials are considered textured when the orientation of grains is not random. Otherwise, the material is not textured. The textured state of a material could be viewed as an intermediate state in between a completely randomly oriented polycrystalline powder and a completely oriented single crystal. The determination of orientation in grains of a polycrystalline sample is important to describe texture. Various processes such as deformation of metals and oxidation in materials are better understood using texture. Anisotropy of various materials' properties, such as plastic deformation, various mechanical properties and corrosion and oxidation are caused by texture.

There are several experimental methods that can be used to measure texture. Neutron diffraction is one of the most commonly used. The result of texture measurement obtained using the neutron diffraction method, is represented by a pole figure, which is a stereographic projection of pole density as a function of pole orientation [4].

In this work, the texture or the distribution of preferred orientation in Fe-Ni LCS is investigated, because the texture is related to the changes in the mechanical properties (i.e. residual stress) of the steel.

EXPERIMENTAL METHOD

Materials

An austenitic low Carbon (A1) with a non-standard compositions of 20.0Ni, 21.0Cr, 1.5Si, 2.0 Mn, <0,085C 0,08Ti in mass percent was used in this study. Both XRD and neutron diffraction analysis confirm the cubic *Fm3m* space group and the FCC cubic unit cell pattern of the sample with a cell parameter of a = 3.564 Å. The samples were available in both as-cast and hot rolled conditions. The samples are further prepared in the shape of bars of approximately 22 cm in length and 2.5 cm x 2.5 cm in cross section, to be used as specimens in the neutron diffraction texture experiments. The samples are then cut in half to obtain the as cast and rolled samples. The bar shaped samples are shown in Figure 1. Fe(Ni- LCS) samples are hot rolled at 1200 °C and 40% reduction in the P2M lab LIPI.

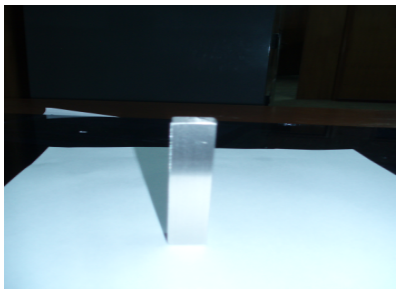

Figure 1. A1 bar sample

Table 1. Conditions of neutron measurement using the residual stress measurement instrument PD DN1 (stress measurement system).

Condition \ Facility	Neutron Scattering Laboratory of Center for Technology of Nuclear Industrial Materials of National Nuclear Energy Agency (BATAN), Indonesia
Instrument	RSM sistem
neutron wavelength	0.183404 nm
Incident slit	5 mm × 5 mm (residual stress) Open F1 (texture)
Receiving slit	5 mm × 5 mm (residual stress) 150 150 (texture)

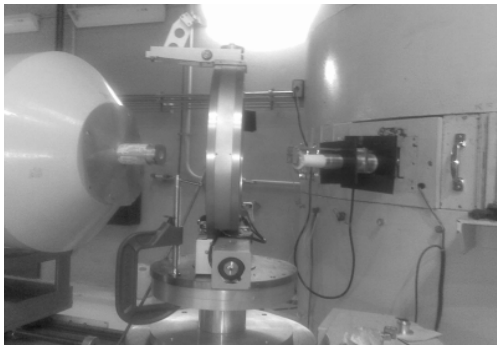

Figure 2. ND test setup

Measurement

Texture measurements were performed using the DN-3 Powder Diffractometer located at the Neutron Scattering Laboratory RSG-GAS of National Nuclear Energy Agency (BATAN), Indonesia. In Table 1, the experimental conditions for the residual stress and texture measurements using the PD DN1 facility are listed.

In Figure 2 the experimental arrangement showing the X,Y,Z translator and the specimens is shown. In Figure 3 the geometrical arrangements defining the measurement angles ϕ, ψ or χ is shown. The scanning ϕ and χ was performed at 5° intervals, and the measured ranges were from -90° to 90° and from 0° to 90°. In A1 alloys, three sets of crystal planes have Bragg reflection intensities that are strong enough to be monitored for the production of a pole figure. The Miller indices are: (111) the strongest, (200) of lesser intensity and parallel to the crystal a-axis, and (220) also of lesser intensity but parallel to the b axis.

Data Analysis

The texture could simply be described by its ideal orientation, i.e. (*hkl*) [*uvw*], provided the ideal orientation in a polycrystalline material could be identified. Nonetheless, this is not a fully quantitative description and in most cases, texture can not be represented by one ideal orientation. To provide a quantitative description of texture the so called ODF

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(Orientation Distribution Function) is used, and is defined in Equation 1.

$$dV/V = f(g)dg \quad \text{.....} \quad (1)$$

Where :

f(g)= ODF
dV = Volume of grains of orientation g within the element of orientation space dg and
V = Volume from which data are collected.

The pole figures describing texture of the samples were generated by calculating quantitative orientation distribution functions (ODF) with the help of MAUD application code developed earlier by Lutterotti [5]. A pole figure is a projection in two dimensions (2D) of the ODF for a specific (*hkl*) plane. An orientation distribution for any given axis inclined at an angle θ relative to any given direction can be expressed as a Legendre polynomial of order n, if the distribution of crystallographic axes is assumed to have cylindrical symmetry about the principal processing directions, as Equation 2.

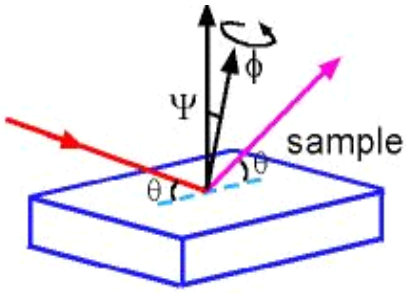
$$< P_n > = \frac{\int_0^{\pi/2} I(\theta)\sin(\theta)P_n(\cos\theta)d\theta}{\int_0^{\pi/2} I(\theta)\sin\theta d\theta} \quad \text{.....} \quad (2)$$

For n=2, the Legendre polynomials of Equation (2) reduces to the well known ″Hermanns″ orientation function, also simply referred to as P2,

$$< P_2 > = P_2(\cos\theta) = \frac{1}{2}(3\cos^2\theta - 1) \quad \text{.....} \quad (3)$$

The three principal processing directions: the direction along the length of the process line or machine direction (roll direction) (MD), the direction across the width of the process line or transverse direction (TD) and the normal direction (ND), have been related to the three principal crystalline axes of the microcrystallites by some authors.

The discrete texture methods use Entropy WIMV (derived from WIMV). The ODF space described in the three eulerian angles is divided in small cells each one defining the value of the ODF in its volume. The pole figure value for each peak of each spectrum is computed by a numerical integral throughout the ODF space considering a tube projection or averaging over adjacent cells. In MAUD an external cycle performed once every refinement cycle extracts the pole figure values from the spectra using a tuned Le Bail algorithm and computes the ODF through an entropy method. Higher order ODFs are expressed in terms of generalized spherical harmonics, an *n*th order three dimensional series expansion. The highest order of the expansion, *n*, is determined by the



Pole Figure Measurement

Figure 3. Coordinate system and defining the angles *Φ, Ψ* OR *X*

symmetry of the unit cell (orthorhombic for PE), the symmetry of the sample (orthorhombic for a film), and the amount of data (e.g. number of pole Figures = 3 [6].

$$w(\theta,\phi,\eta) = \sum_{l=0}^{\infty}\sum_{m=-l}^{l}\sum_{n=-l}^{l} W_{lmn} Z_{lmn}(\cos\theta)e^{-im\phi}e^{-in\eta} \quad (3)$$

The resulting W_{rmn} or "Roe-Krigbaum" coefficients are the *lmn*th moments of the ODF, which result directly in the f_{lmn} orientation factors.

$$f_{lmn} = 4\pi^2\left[\frac{2l+1(l+m)!(l+n)!}{2(1-m)!(1-n)!}\right]^{-1/2} W_{lmn}$$

RESULTS AND DISCUSSIONS

Figure 4, shows the results of a -2θ scan of a hot rolled A1 LCS taken in the [111] crystallographic direction.

In Figure 5 the full pole figure for the as cast (nonroll) LCS specimen in mirror symmetry and ODF relation of 10° is shown. Pole densities are expressed in terms of mrd (multiple random distributions) unit. The pole figures in Figure 5 and Figure 6 reveal that the as cast machined and rolled plates show a significant texture. After corresponding rotations, pole figures derived from as cast machined and rolled plates are quite different (see Figures 5 and 6). The main feature is a minimum in the [111] pole in the circumferential direction as indicated

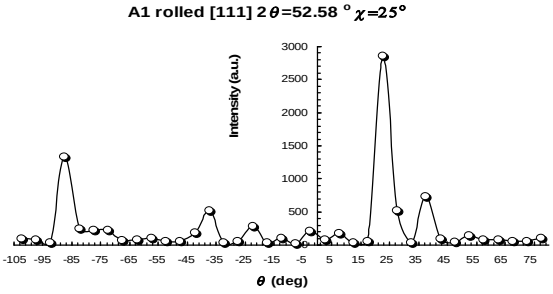


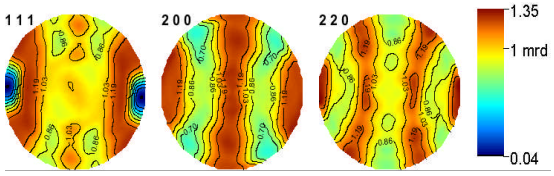
Figure 4. θ-2θ scan of a hot rolled A1 LCS taken in the [111] (2θ=52.58°) crystallographic direction and χ=25°

by the darker spots at 9 and 3 o'clock in Figures 5, but in the [200] and [220] pole directions (p.d.) these locations are maximum spots, and in the [200] pole direction the maximum runs through the central equator. In the addition, irregular texture pattern occur in the [111], [200] and [220] pole parallel to all three primary directions (drawing, circumferential, and radial) as indicated by the Figures 5, p.d. [111], at 3 and 9 o'clock, central equatorial, and centre, respectively. Pole figures of the hot-rolled bar show stochastic variations due to poor grain statistics (see Figure 6). A more regular pattern becomes apparent after additional analysis by narrowing the ODF resolution to 5°, a maximum intensity of 2.01 mrd could be observed as in Figure 7, but the distribution is very asymmetric.

Scattering of the data in Figure 5 is attributed to recrystallization and grain growth experienced during the annealing [7]. Figure 5 shows that the as cast steel bar texture is generally not the same as the hot rolled bar at 1200 °C, although the degree of texturing was slightly less in the high temperature hot-rolled condition, as indicated by the lower pole densities (the same contouring levels were used for all pole figures).

In Figure 6 the full pole figure for the hot rolled LCS specimen in mirror symmetry and ODF resolution of 10° is shown.

In Figures 8 and 9, the partial inverse pole figure for the as-cast (nonroll) and the hot-rolled A1 LCS specimens are shown. These are inverse because instead of using the sample axes as the reference frame the crystal axes become the reference frame. These inverse pole figures show where the rolling direction (1), transverse direction (2), and normal direction (3) appear in reference



Gambar 5. Pole figure for the as-cast (nonroll) A1 LCS specimen

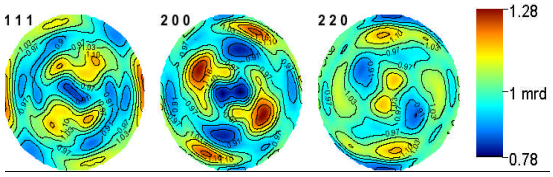


Figure 6. Pole figure for the hot-rolled A1 LCS specimen

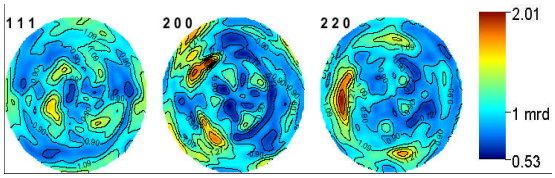


Figure 7. Pole figure for the hot-rolled A1 LCS specimen ODF resolution of 5°

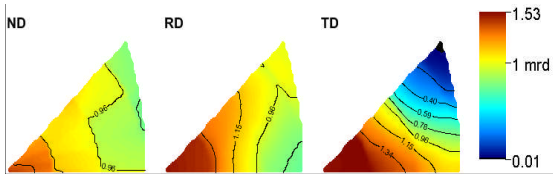
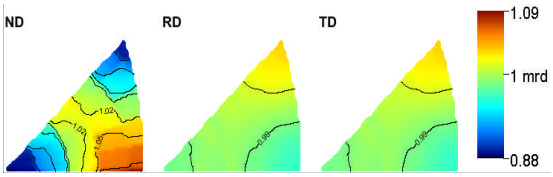


Figure 8. Partial inverse pole figure for the as-cast (nonroll) A1 LCS specimen



Gambar 9. Partial inverse pole figure for the hot-rolled A1 LCS specimen

to the crystal axes. One plot is for one sample axis. For the as cast steel bar, the maximum pole densities occur in all three axial directions, with the transverse direction (TD) showing the most variations in pole densities. The texture variation narrowed in the hot rolled bar, and maximum pole density occurs only in the normal direction (ND). Consequently, by comparing the two figures it could be concluded that hot rolling has affected the preferred orientation distribution or the texture of a A1 LCS. While the maximum intensity (1.35 mrd and 1.28 mrd respectively) has not changed significantly in both cases, the minimum intensity and the ratio of maximum and minimum have changed quite significantly. Also the positions of the concentric-circular distribution have been altered after hot rolling. However, no areas of very high pole density could be found in both cases, and the highest density is only about 1.38 times the mrd, indicating a weak texture in the materials.The pole figures for this plate are consistent with face-centered cubic (FCC) rolling textures [6,8].

CONCLUSIONS

The ODF plots for the A1 as cast and 1200 °C hot rolled specimens show typical patterns for recrystallized and rolled cubic structure.

It is concluded that these pole figures are consistent with FCC rolling texture.

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