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# AN X-RAY METHOD FOR THE DETERMINATION OF STORED ENERGIES IN TEXTURE COMPONENTS OF DEFORMED METALS; APPLICATION TO COLD WORKED ULTRA HIGH PURITY IRON

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**Abstract**—An X-ray method has been developed to evaluate the stored energy of cold work in different texture components of plastically deformed metals. The dislocation density and the outer cut-off radius of dislocations are obtained from Bragg peaks recorded from single texture components. The stored energy is approximated by the energy of dislocations, which is calculated according to the anisotropic theory of elasticity. As an example the method is applied to the case of two major texture components developed in cold rolled ultra high purity (UHP) iron. The stored energy of the  $\{111\}\langle 112\rangle$ - $\gamma$  fibre component of the 88% cold rolled UHP iron is about 3.6 times larger than that of the  $\{001\}\langle 110\rangle$ - $\alpha$  fibre component. The present results, of significantly higher accuracy than those of previous methods, are in good agreement with data obtained from microhardness and recent calorimetric measurements. © 2000 Acta Metallurgica Inc. Published by Elsevier Science Ltd. All rights reserved.

Keywords: Cold working; X-ray diffraction; Dislocation density; Texture; Iron

## 1. INTRODUCTION

During cold work of a metal, a small fraction of the energy of deformation is stored in the crystal in the form of the elastic energy associated with the strain field of the generated dislocations. Grains with different orientations experience different amounts of slip and different types of slip interactions and consequently store different amounts of energy after deformation. To evaluate this orientation dependent stored energy, previous works have mainly used X-ray [1, 2] or neutron [3] peak broadening methods. For example, in low carbon steels the following sequence of stored energies was found for grain orientations parallel to the rolling plane:  $W_{(110)} > W_{(111)} > W_{(112)} > W_{(100)}$  [1]. Similar results have been obtained using electron microscopy data for size and subgrain misorientations in Ref. [4]. The stored energy is the driving force for recrystallization; its orientation dependence is known to influence the development of recrystallization textures, which are very important for industrial applications.

A general feature of the diffraction methods mentioned above is the application of the Stibitz formula which relates the stored energy to the mean change in lattice spacings [5]. This formula, obtained under the assumption of an elastically isotropic material with random distribution of principal residual-stress axis, is rather difficult to apply for the case of elastically anisotropic and/or textured materials, as often encountered in practice. Its application is also rendered inaccurate by the *strain anisotropy* effect caused by dislocations [6]. Owing to all these factors, it seems that a more accurate evaluation of the stored energy can be made by considering the energy of dislocations present in the crystal.

Usually the final dislocation structure of a cold worked metal is heterogeneous, its total energy is approximated by the sum of the following terms [7]:

$$W = W_{\rm disl} + W_{\rm het} + W_{\rm mean} \tag{1}$$

where  $W_{\rm disl}$  is the energy of dislocations,  $W_{\rm het}$  is the energy arising from their heterogeneous distribution, e.g. the elastic energy associated with the long-range stress fields in cellular dislocation structures [8–10] and  $W_{\rm mean}$  accounts for the

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polycrystalline nature of the material representing the elastic energy contribution of the mean stresses developed in single grains of the polycrystal due to the heterogeneity of deformation at the grain level. Both long-range and mean stresses can be described by the assumption of geometrically necessary dislocations which account for strain compatibility between adjacent regions. As pointed out for copper [7]  $W_{het}$  is about one order of magnitude smaller than the contribution of dislocations and  $W_{\text{mean}}$ < 10% for grain sizes > 30  $\mu$ m [11]. Thus, to a first approximation, the stored energy of a plastically deformed metal can be considered equal to the total energy of ordinary dislocations. This is the basis of the new evaluation method presented here, which considers the density and the outer cut-off radius of dislocations determined from Bragg peak-profiles and the elastic anisotropy of the investigated material. As an example, the case of two major texture components developed in cold rolled UHP iron will be treated in detail.

#### 2. EXPERIMENTAL

#### 2.1. Sample characterization and mechanical tests

The UHP iron produced at the École des Mines de Saint-Étienne had a concentration of impurities <1 ppm for 70 investigated elements and was under the detection limit of 10 ppm for Si, and 5 ppm for N [12]. Polycrystalline samples of about 150  $\mu$ m grain size and initial nearly random texture were deformed in plane strain compression to reductions of 48% and 62% after which cold rolling was applied in order to attain a final reduction of 88%. The adopted sample and crystal coordinate systems are indicated in Fig. 1. The polycrystal is compressed along the  $-X_3$  direction and constrained to extend along  $X_1$ , the imposed strain rate components are  $\dot{\varepsilon}_{22} = 0$ ,  $\dot{\varepsilon}_{11} = -\dot{\varepsilon}_{33} = \dot{\varepsilon}$  and  $\dot{\varepsilon}_{23} = 0$ . The plane strain compression tests were carried out at room temperature using a lubricated channel die arrangement in a mechanical testing machine, oper-



Fig. 1. Schematic drawing of the experimental set-up for plane strain compression, together with the sample and crystal coordinate systems. ND represents the normal direction, RD the rolling direction and TD the transverse direction. The surface A was cut at 45° in order to measure the lateral face of the {001}{110} texture component.

ating at a constant strain rate of  $5 \times 10^{-3}$ /s. To reduce friction, a thin (0.05 mm) teflon film was placed around the sample.

After deformation the electropolished samples were placed in a Siemens D500 diffractometer and the {110}, {200} and {112} pole figures were measured using CoK $\alpha$  radiation and an angular step size of 5° in tilt and azimuth. The pole figures were used to calculate the orientation distribution function (ODF) [13] from which the section through the Euler space corresponding to  $\varphi_2 = 45^\circ$  is shown in Fig. 2(a). Mainly two texture fibres are present in the 88% cold rolled UHP iron, the  $\alpha$ -fibre with grains having their (110) along the rolling direction (RD) and the  $\gamma$ -fibre with (111) along the normal



Fig. 2. Section of the ODF corresponding to the Euler angle  $\varphi_2 = 45^\circ$ . (a) Sample cold rolled at 88% reduction and (b) ODF obtained from crystal plasticity simulations.

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