DEPENDENCE OF GRAINS HARDNESS ON THEIR ORIENTATION IN ELECTRICAL STEELS

Petra GAVENDOVÁ[a], Ivan PETRYSHYNETS[b], Martin SOPKO[b], František KOVÁČ[b]

[a]*Brno University of Technology, Faculty of Mechanical Engineering, Institute of Materials Science and Engineering, Brno, Czech Republic, EU, gavendova@fme.vutbr.cz*

[b]*Slovak Academy of Science, Department of Materials Research, Kosice, 040 01, Slovak Republic, EU, ipetryshynets@imr.saske.sk, msopko@imr.saske.sk, fkovac@imr.saske.sk*

Abstract

An investigation of the dependence of grains hardness on their orientation in electrical steel is presented in this work. The study was performed by means of nanoindentation experiments using Nanoindentor G 200 with calibrated Berkovich indenter. Nanoindentation measurements were made at room (26 °C) and at elevated temperatures (100 °C, 200 °C and 250 °C). Nanohardness measurements were carried out in non-oriented electrical steel with columnar microstructure, in order to evaluate local variation of work hardening as a function of crystallographic orientation. The grain orientation with respect of individual rolling planes and rolling orientation was determined by EBSD (Electron Back Scattered Diffraction) analyses. An EBSD analysis was used also determine grain orientations, in which were made the nanohardness measurements. Indentation hardness H_{IT} was determined from load-displacement curves within individual grains (G1, G2, G3) with various crystallographic orientations. Hardness was shown to decrease with increasing temperature in each of individual grains. The differences of hardness values were observed also between particular grain orientations. It is shown that for a specific deformation, the behaviour of each grain is determined only by its current orientation and slip system.

Keywords: Nanoindentation technique, nanohardness measurement, elevated temperature, crystallographic orientation

1. INTRODUCTION

Nanoindentation has proven to be an effective and convenient method of determining the mechanical properties of solids, most notably elastic modulus and hardness due to its simplicity and easiness [1, 2]. It records the indentation depth continuously with indentation load, while conventional hardness tests observe the residual imprint as a contact area using an optical microscope [3]. During nanoindentation, a diamond indenter is pushed into test material and then withdrawn. The indentation load and the penetration depth into the material are simultaneously recorded and a load-penetration depth (F-h) curve is obtained. This F-h curve contains a wealth of information relating to the deformation behaviour of materials and can be used to determine many mechanical properties such as hardness [4]. Hardness is usually defined as the mean contact pressure between the indenter and the specimen, and it has been used for qualitative comparison of material strengths. The interpretation of indentation load-depth curve is very ambiguous because of complex indentation stress fields beneath the indenter. In addition, hardness itself is not a basic material property for indicating the material strength [5, 6]. In other words, hardness is affected by the elastic and plastic properties of material, the indenter sharp, and partially by the experimental procedure and the surface condition of specimen.

Nanoindentation at high temperature or elevated temperature presents an additional capability in nanoindentation techniques, which have demonstrated tremendous potential in the study of nanoscale mechanical behaviour. At elevated temperatures, however, the unloading response associated with many different classes of materials during nanoindentation becomes viscoelastic in nature, and the conventional analyses of the load-depth curves are no longer valid [7]. The present work considers the dependence of

grains hardness on their crystallographic orientation using nanoindentation technique at room and at elevated temperatures. A commonly used method for analysing mechanical properties such as hardness from P-h curves is that proposed by Oliver and Pharr [8].

2. EXPERIMENT

As experimental material, vacuum degased non-oriented (NO) electrical steel after final annealing in laboratory conditions was used with dimension of 2x4 mm. The chemical composition of investigated material is presented in **Table 1**. The microstructure of the studied material is shown in **Fig. 1.** As one can see, the average grain size of this steel is in the range 150 - 250 μm.

Table 1 Chemical composition of the investigated steel (wt %)

Steel	C (%)	Mn(%)	Si (%)	Cu (%)	P (%)	S (%)	Al (%)	N (%)	Ti (%)
M 340	0.0036	0.183	1.25	0.010	0.039	0.001	0.128	0.0048	0.004

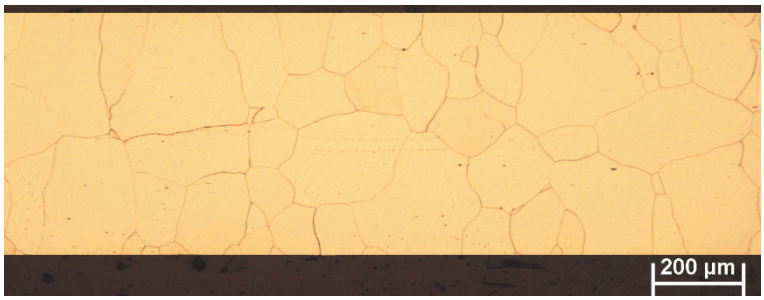

Fig. 1 Microstructure of NO electrical steel

The crystallographic orientation of grains with individual orientations was examined by means of Electron Backscatter Diffraction method. The analysis was performed mostly on the longitudinal cross section of the samples. Nanoindentation experiments were performed by Nanoindentor G 200 instruments equipped with calibrated Berkovich indenter at the maximum force of 25 mN. Loading as well as unloading process were carried out during the twenty seconds with holding time ten seconds. The laser heating system of instrument allows heating realization of sample and the tip independently. The measurements were performed at 26 °C, 100 °C, 250 °C and 250 °C for the each grain. A standard Continuous Stiffness Measurements (CSM) method was used for measurements at room temperature.

At elevated temperatures a modified CSM method was used instead. This method includes so-called stabilization time (about 300 second), which provide the tip contact with sample surface before indentation process. The sample was fixed to the heated holder using a special cement paste. Indentation hardness H_{IT} was determined from load-displacement curves by means of Oliver-Pharr method [8]. The presented data here were acquired during a single continuous heating sequence. Each grain was measured at target temperature after stabilization time (about 2 hours). The sample was heated to the next temperature, without intermediate cooling, when all grains were measured at specific temperature. The specimen was cooled down to the room temperature, when the last measurements were finished on the selected grains at 250 °C. The heating as well as the cooling rate was 1.6 °C/min.

3. RESULTS AND DISCUSSION

The investigated grains of the examined materials are characterized by single crystal orientation in space with low defects (dislocation) density. **Fig. 2** presents the IPF (Inverse Pole Figure) map which describes the crystallographic orientation of detected grains of silicon steels. In order to study the differences between crystallographic orientation and nanohardness measurements, three grains with different crystallographic orientations in the sheet plane were chosen; G1 {111}//ND, G2 {001}//ND, G3 {011}<001>. These grains were subjected to the nanoindentation measurements. The twenty indentations have been carried out to the selected grains at mentioned temperatures in array of 2x10 with spacing of 25 μm. **Fig. 3** shows the residual indents in the grain G1 with {111}//ND crystallographic orientation.

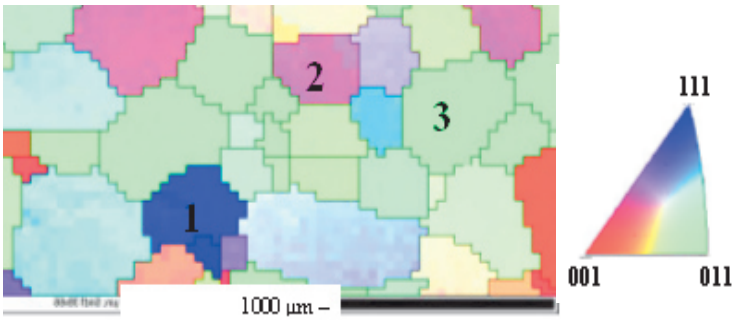

Fig. 2 IPF map representing the grain of NO steel

Fig. 3 The indentation array at G3 after room and elevated temperatures measurements

The results obtained by nanoindentation tests at room temperature and elevated temperatures are presented in **Figs. 4** and **5**, respectively. **Fig. 4** shows the dependence of grains hardness on penetration of nanoindentor tip into the sample surface. The color of curves shows the main texture components of grains. The yellow, red and green graphs represent the grains G1, G2 and G3 with various crystallographic orientations. The higher value of hardness was measured on grain G1 with {111}//ND orientations and the lower hardness values was obtained on grain G2 with {001}<001> and then on G3 with {011}<001> orientation. Although the mentioned values of the magnitude differ from each other in quite small scale, the denoted results clearly showed that, accumulated energy in grains very depends on them orientation [9].

The typical nanoindentation results of NO steel are illustrated in **Fig. 5**. From the loading portion of the F-h curve we observed that the maximum depth of the indentations does not change very much at temperature increase up to 100 °C. Further increase to 200 - 250 °C leads to a sudden increase in the indentation depth.

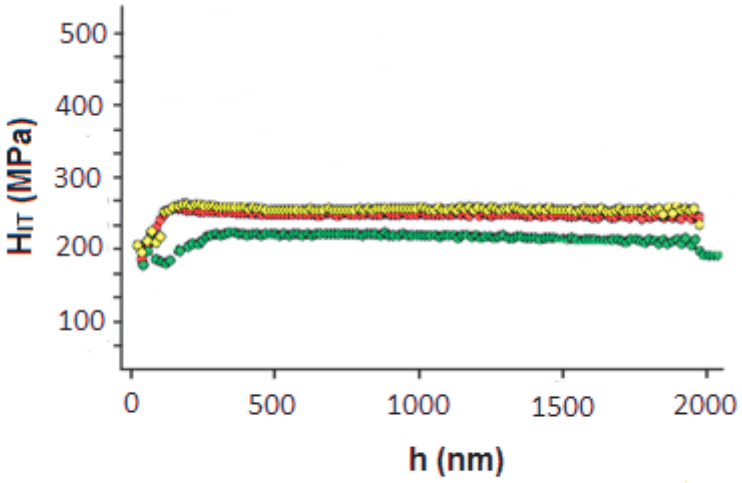

Fig. 4 Dependence of grain H_{IT} on depth at room temperature

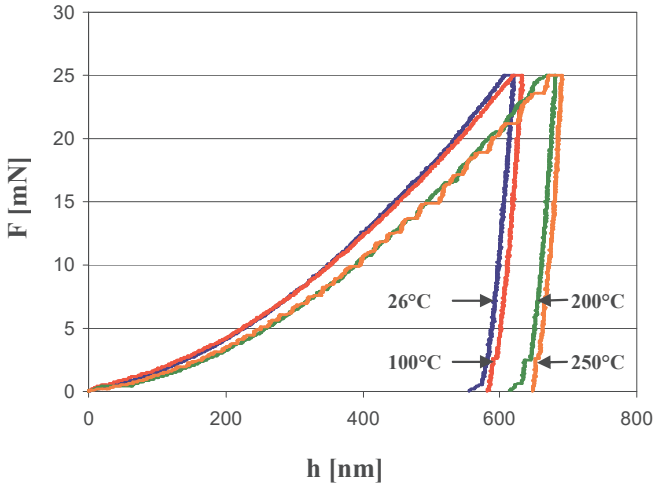

Fig. 5 Individual F-h curves obtained by nanoindentation of NO steel at elevated temperature

Fig. 6 shows the results from nanohardness H_{IT} measurements. As one can see, indentation hardness slightly decreases with increasing temperature for grain G1 from room temperature up to 250 °C from 2,9 GPa to 2,3 GPa, for grain G2 from 2,8 GPa to 2,1 GPa and for grain G3 for 2,7 GPa to 2,1 GPa. Some differences in hardness values are also between particular grain orientations. Decrease of hardness with increasing temperature for grains G1, G2 and G3 was about 20%, 26% and 22% respectively. This differences between individual grains with various orientation, can be related to various number of active slip system in grains [10].

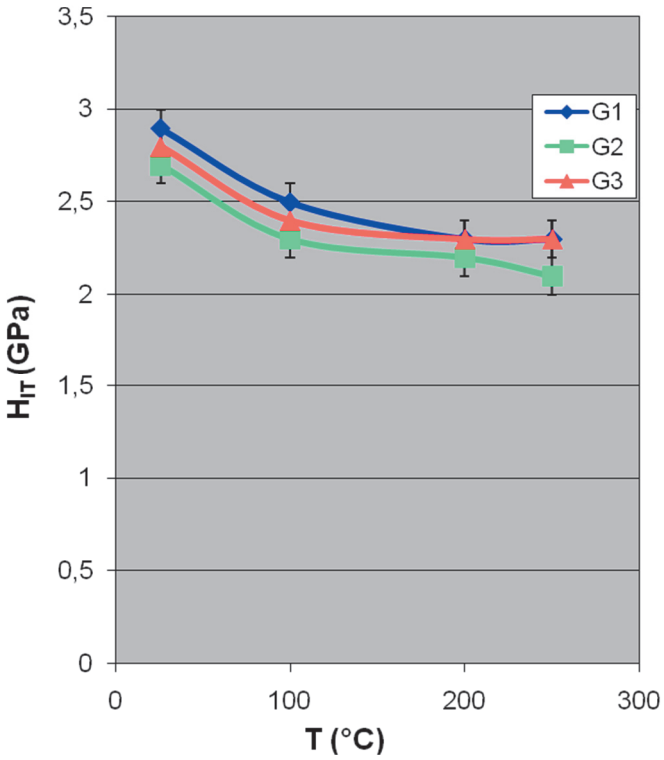

Fig. 6 Nanohardness results for each grain

CONCLUSION

In situ room and high temperature nanoindentation experiments were performed in order to study the dependence of grains hardness of their orientation. On the basis of results from nanoindentation measurements in each of particular grain with different crystallographic orientation it was observed that the hardness was found decrease with increasing temperature and the differences of hardness values were observed also between particular grain orientations. Decrease of hardness with increasing temperature for grains G1, G2 and G3 was about 20%, 26% and 22% respectively. This can be explained with various number of active slip system in the individual grains.

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