NANOHARDNESS OF GRAINS OF ELECTROTECHNICAL STEEL AT ELEVATED TEMPERATURE

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Abstract
The differences in nanohardness H_{IT} between particular grains of non-oriented electrical steel at room and elevated (100, 200, 250 °C) temperature were observed using the nanoindentation technique. The elevated temperature indentation response was examined using an indentation Nano Test NT 600 system modified with tip and sample heating. Indentation hardness reveals descending tendency with increasing temperature in each of investigated grains with crystallographic orientations in the sheet plane – G1 {111}//ND, G2 {001}//ND, G3 {011} <001>. The indentation hardness showed some differences in values of indentation hardness between particular grain orientations. In the temperature range of 200-250 °C the load-depth (F-h) curves reveal the pop-in effect.

Keywords: non-oriented electrotechnical steel, high temperature nanoindentation, nanohardness, grain orientations

1 Introduction

Non-oriented (NO) electrical steel sheets are tailored to produce specific magnetic properties. The most important requirements are a high permeability and a low magnetic core loss. Their magnetic properties depend critically on microstructure and its components, e.g. grain size, content of non-metallic inclusions, surface and texture [1-3]. For non-oriented electrical steel (C under 0,02 wt.%) it is possible to apply a decarburizing annealing in the intercritical region that leads to the columnar-grained microstructure with a specific type of the texture, thus avoiding the heterogeneity [4, 5]. By application of such an annealing it is possible to enhance desirable so-called “random cube” {100} <0vw> texture component in the final material, which cannot be obtained by applying ordinary rolling and annealing processes [6, 7].

For vacuum degassed electrotechnical steel (C under 0,006 wt. %) we used the process of strain induced grain boundary migration (SIGBM) for the growth of columnar grains [8, 9]. Study of deformation mechanism has evolved in order to explain precauses behind the changes of microstructure and texture during deformation. Change in orientation and shape of individual grains in polycrystalline material is caused by plastic flow due to generation of dislocations moving in certain direction (slip directions) within certain planes (slip planes) [10-12].

Instrumented indentation testing or nanoindentation has emerged as an important method for evaluation of material´s mechanical response to applied loading of thin films and surface layers of submicron thickness [13-16]. The most popular method relies on an analysis of the unloading load-displacement response which is assumed to be elastic even if the contact is elastic-plastic [8, 17]. The most common method for evaluation of hardness from the load-displacement data obtained with Berkovich indenter was proposed by Oliver and Pharr [18].

High-temperature or elevated temperature nanoindentation testing presents an additional capability in nanoindentation techniques and has also been applied to study a wide range of temperature-sensitive materials phenomena, including temperature-dependent mechanical properties, deformation mechanisms, rheology and phase changes [19-21]. 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-displacement curves are no longer valid [22]. In this study nanoindentation tests were conducted for comparison of nanohardness between the particular grain orientations (with (001); (011); and (111) planes perpendicular to the loading) at room and elevated temperature.

2 Experimental material and methods

The sample of NO electrical steel were taken from industrial line after hot rolling and subsequently annealed in laboratory conditions at 900°C/5min. The annealing atmosphere was pure hydrogen (d.p. ~ -23 °C). The thickness of the steel was 1, 8 mm. The chemical composition of investigated material is presented in **Table 1** and microstructure of the studied steel is presented in **Fig. 1**, with the average grain size about 150-250 µm. Crystallographic orientation of single grains was examined using Electron Backscatter Diffraction (EBSD) commonly used for microstructural-crystallographic analysis.

Table 1 Chemical composition of investigated NO steel

Element	C	Mn	Si	P	S	Al	N
%wt	0.004	0.23	2.8	0.008	0.005	0.48	0.004

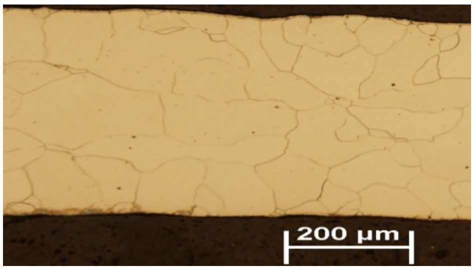


Fig. 1 Microstructure of investigated NO steel

Nanohardness H_{IT} measurements was carried out at room and elevated temperatures (100, 200, 250 °C) using NanoTest NT 600 instrument equipped with the calibrated Berkovich indenter and load control system. Loading as well as unloading lasted 20 s with the maximum force of 25 mN and the hold period was 10 s. The NanoTest system was equipped with a computer-control heating stage for measurement at elevated temperatures. Heating was applied to both indenter and the sample utilizing separate temperature control. This isothermal contact ensures that there is no heat flow during indentation process. The sample of dimension 2x4 mm was fixed to the heated holder using a special cement paste [23].

Nanohardness H_{IT} was determined from load-depth curves using Oliver and Pharr method [18, 24]. The presented data here were acquired during a single continuous heating sequence. Each grain was measured at target temperature after stabilization (typically 2 hours). When all grains were measured at specific temperature the sample was heated to the next temperature, without intermediate cooling. When all of the experiments were completed, the specimen was cooled down to room temperature. Heating as well as cooling rate was 1.6 °C/min.

3 Results and discussion

Investigated grains of the examined material are characterized by single crystals orientation in space with low dislocation density. Inverse Pole Figure (IPF) map obtained from the sample of **Fig. 1** is presented in **Fig. 2**. This map is used to define the crystallographic orientation of grains in the investigated material.

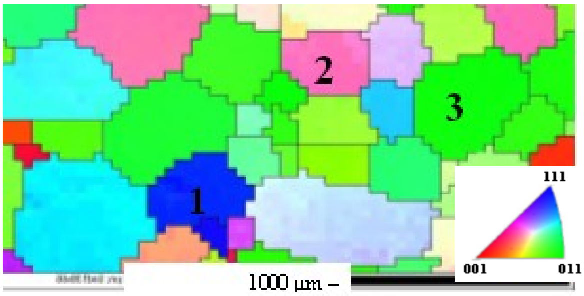


Fig. 2 IPF map representing the grains of NO steel

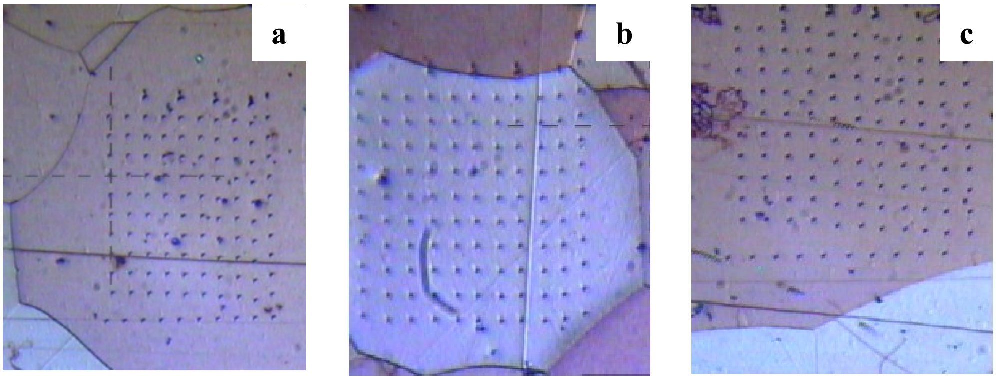
In order to study the effect of crystallographic orientation on indentation properties three grains with different crystallographic orientation were chosen; G1 {111}//ND; G2 {001}//ND; G3 {011} <001>. These grains were subjected to the nanoindentation measurements. 20 indentations have been carried out for each grain at each temperature (room temperature, 100 °C, 200 °C, 250 °C) in array of 2x10 with spacing of 25 µm. **Fig. 3a-c** shows the residual indents in the grains G1, G2 and G3.

Fig. 4 shows the indentation load – depth (F-h) curves of the NO electrical steel at room and elevated temperatures. There we observe that the maximum depth of the indentations does not change very much as temperature is increased to 100 °C, but then at 200-250 °C there is a rather sharp increase in indentation depth and we observe that there is a prominence of pop-in effect [25]. This process is related to a sudden displacement jump in the load-depth curve. The pop-in effect was observe in all three grains and according to Johansen et al.[25] this effect could be related to homogeneous nucleation of dislocation during nanoindentation.

From the loading-unloading curves as shown in **Fig. 4,** indentation hardness H_{IT} can be obtained by Oliver and Pharr model [17]. The Analyzes of load depth (F-h) curves showed the pop-in effect (the jump in the load-depth curves) during elevated temperature nanoindentation. The pop-in effects were observed at 200 – 250 °C and could be related with homogeneous nucleation of dislocation during nanoindentation.

Fig. 5 shows that the value of hardness decreased as temperature increased. The value of the hardness slightly decreased from 2.7 GPa at 26 °C to 2.3 GPa at 250 °C in grain G1, from 2.7 at

26 °C to 2.1 at 250 °C in grain G2 and from 2.8 at 26 °C to 2.1 at 250 °Cin grain G3. As one can see in **Fig. 5** there are some differences in H_{IT} values between particular crystallographic planes. Decrease of hardness with increasing temperature for grains G1, G2 and G3 was about 15%, 22% and 25 % respectively.



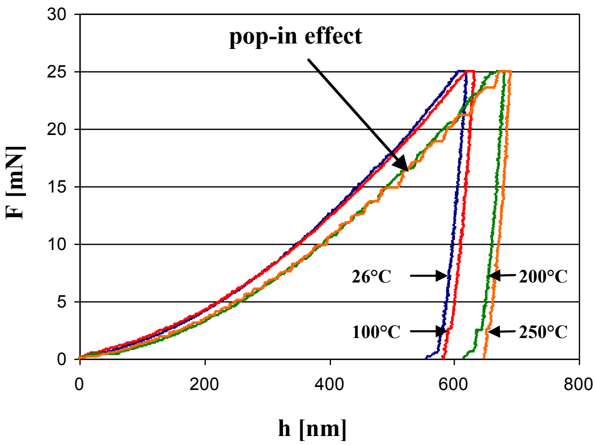


Fig. 3 Load-depth (F-h) curves obtained from NO electrical steel at various temperature

These differences between individual grains can be related to various number of active slip systems in corresponding orientations [26]. A decreasing trend in hardness with increasing temperature using a Berkovich indenters in load control have been observed [20, 27].
The results of indentation hardness H_{IT} from nanoindentation at room (26 °C) and elevated temperatures (100 °C, 200 °C, 250 °C) are summarized in **Table 2**.

Table 2 Results of H_{IT} values from nanoindentation at various temperatures

Element	**26 °C**	**100 °C**	**200 °C**	**250 °C**
G1 H_{IT} (GPa)	2.7±	2.5±	2.3±	2.3±
G2 H_{IT} (GPa)	2.7±	2.3±	2.2±	2.1±
G3 H_{IT} (GPa)	2.8±	2.4±	2.2±	2.1±

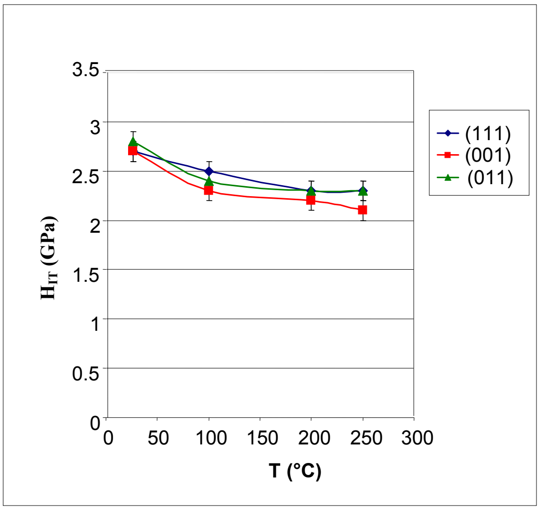


Fig. 4 Dependence of hardness on various temperature and crystallographic plane

4 Conclusions

Nanohardness H_{IT} of the non-oriented electrical steel in particular grain orientations have been studied by nanoindentation technique, at various temperatures (26 °C, 100 °C, 200 °C, 250 °C). The Analyzes of load depth (F-h) curves shows the pop-in effect (the jump in the load-depth curves) during the nanoindentation at elevated temperatures. The pop-in effect was observed at 200 – 250 °C and could be related to homogeneous nucleation of dislocation during the nanoindentation.

The results from hardness H_{IT} measurements show that the nanohardness for all investigated grains G1, G2, G3 decreased as temperature increased from room temperature up to 250 °C. The differences of H_{IT} values were also observed between particular grains of different orientations and this can be explained by various number of activated slip system in the individual grains.

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