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EFFECT OF VC PRECIPITATES ON THE FORMATION OF MICROSTRUCTURES AND MAGNETIC PROPERTIES OF GO ELECTRICAL STEELS

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Some new procedures of grain-oriented (GO) steels processing have been used in the present work. The suggested approach combines an application of new systems vanadium carbide (VC) nano - particles, temper rolling processes and the dynamic continuous annealing for the secondary recrystallization in the investigated steels. The experimental grain-oriented steels with proposed systems VC inhibitors and at state after temper rolled were subjected to dynamical (fast heating) annealing in order to obtain abnormal grain growth with Goss crystallographic orientation and considerably reduce the preparation time so that the whole process lasts only several minutes. The EBSD analysis shown that suggested procedure led to evolution of the sufficiently strong {110}<001> Goss texture, which is comparable to that obtained in the conventionally treated GO steels. Moreover, the steels treated by the newly developed method showed similar magnetic properties as the material which was passed after the long-time heat treatment. The achieved coercive field values of the laboratory treated samples are nearly identical (AC conditions) or even slightly lower (DC conditions) to those of reference samples taken after industrial final box annealing.

Keywords: GO silicon steel, magnetic anisotropy, coercivity, texture analysis, microstructure

1 INTRODUCTION

The earliest soft magnetic material was iron, which contained many impurities. Researchers found that the addition of silicon increased resistivity, decreased hysteresis loss, increased permeability. Grain oriented (GO) steels are iron 3% silicon alloys developed with a sharp {110}<001> texture, also called the Goss – type texture, to provide very low power loss and high permeability in the rolling direction. These steels are predominantly employed for highly efficient transformers [1]. Method of grain oriented silicon steel (GOSS) production invented by Norman P. Goss [2] in the 1930's led to revolutionary reductions in core loss for 3% silicon-iron largely through the development of texture or preferred orientation via the process of secondary recrystallization. The global trend in energy saving and environmental protection has aroused deep interest in the properties and total transformer performance of the GO silicon steels. From that time, this kind of steel has been presented the great interest from both engineering and scientific point of view. The Goss texture ({110}<001>) formation in the mentioned steel represents a particular interest for many researchers through last eight decades [3]. Unceasing investigation of the silicon steels leads to development of two basic types of these materials: conventional grain oriented (CGO) and high oriented or high permeability (HGO) steels. Besides the production route, the main difference between these two types of GO steels is in magnetic properties which very closely related to crystalline orientation. The average deviation of {110}<001> oriented grains from ideal one is about 7 and 3 degrees for CGO and HGO steels, respectively. The mentioned features of the GOSS in turn lead to better magnetic induction in HGO steel, about 1.9 T and 1.7 T in CGO one [4].

The Goss texture is evolved by a secondary re-crystallization that takes place during the long time final box annealing. The steel is typically heated up to 1200ºC with a very low heating rate of 15 – 25ºC/h and subsequently it is annealed at this temperature for around 30 – 40h. The above mentioned process of annealing proceeds for 100 – 120 hours [5]. In order to achieve the {100}<001> texture development during the secondary recrystallization by means of the box annealing it is necessary to provide: (i) inhibitors of the normal grain growth by dispersion of small (50-100nm in size) second phase particle such as MnS, AlN and MnS+AlN, (ii) a presence of the {100}<001> oriented grains in the primary recrystallized fine grained matrix [6].

In the present work we suggest a novel approach for the preparation of the GO steels that consist in an application of nano – particles VC in combination with strain induced grain boundary motion and continuous annealing for the secondary recrystallization. This approach considerably reduces the preparation time as the whole process lasts only several minutes [7]. Special interest was focused to the study of the influence of VC particles on the texture development and the grain growth. The abnormal grain growth thermal activation [8] is supported by the strain induced grain boundary motion. This proposed procedure could lead to a marked reduction of production costs of the grain oriented electrical steels providing energy savings with favorable impact on environment.

2 EXPERIMENTAL PROCEDURE

The material investigated in this work was a GO steel which was prepared in the laboratory conditions with the following chemical composition: C = 0.04, Mn = 0.18, Si = 3.2, P = 0.003, S = 0.003, Cr = 0.008, Cu = 0.54, Al = 0.004, N = 0.003, V = 0.046 wt %. After melting, the obtained slabs which weighed 8 kg with 40 mm thickness were subjected to

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hot rolling process with final rolling temperature 900°C and then subjected to coiling temperatures at 585°C.

Subsequently, strips of steel were subjected to cold rolling with reduction ε~84 % to the thickness 0.35 mm and to subsequent annealing at 1075°C for 10 min. in dry hydrogen atmosphere and to the decarburization process at 850°C for 10 min. in wet atmosphere of H_2 (80 %) + N_2 (20 %) mixed gas (*dp* ~ +40ºC). After decarburization process these samples were temper rolled within one pass. The corresponding thickness reduction was 4 %. Subsequently, the rolled materials were annealed in pure hydrogen H_2 atmosphere upon dynamical heat treatment conditions. The annealing temperature applied to the experimental steel was 1075°C with holding time up to 5 minutes.

The temperature of α/γ phase transformation was detected by means of the differential scanning calorimetry (DSC) analyzer, NETZSCH STA 449 F1 Jupiter.

Carbon extraction replicas for examination of VC particles in material after hot rolling were prepared from metallographic samples and subsequently they were analyzed by means of JEOL 2100F high resolution transmission electron microscope (HRTEM) operated at 200 kV.

The most representative samples were chosen for the microstructure and texture analysis. The texture analysis was carried out by an electron back scattered diffraction (EBSD) method in the normal direction plane. The JEOL JSM 7000F FEG scanning electron microscope was used to perform the texture analysis.

The DC hysteresis loop measurements were performed by Forster type B-H loop tracer.

3 RESULTS AND DISCUSSION

The precipitation parameters of experimental steel were investigated in order to explain the positive impact of VC nano precipitation in the formation of abnormal grain growth with appropriate Goss crystallographic orientation. Observed dispersion of second phase VC particles in laboratory prepared steel was carried out on the samples obtained after hot rolling process and the mentioned coiling procedure at 585°C during 45 minutes in laboratory furnace.

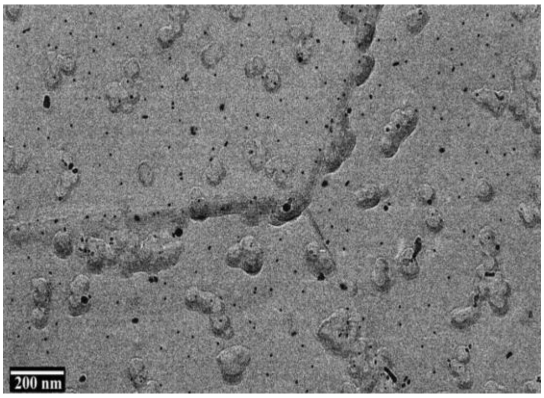

Fig. 1. The distribution of VC nano particles near the grain boundary of experimental steel after the coiling annealing at 585°C and hot rolling

The carbon replica with VC inhibitors is shown in Fig. 1. The identification of these VC particles and their distribution was made by means HRTEM method which included EDX analysis and measurement of distance between the lattice fingers of nano inhibitors at high resolution of TEM, see Fig. 2. As one can see, the obtained sizes of the second phase particles in the investigated material are quite similar. The

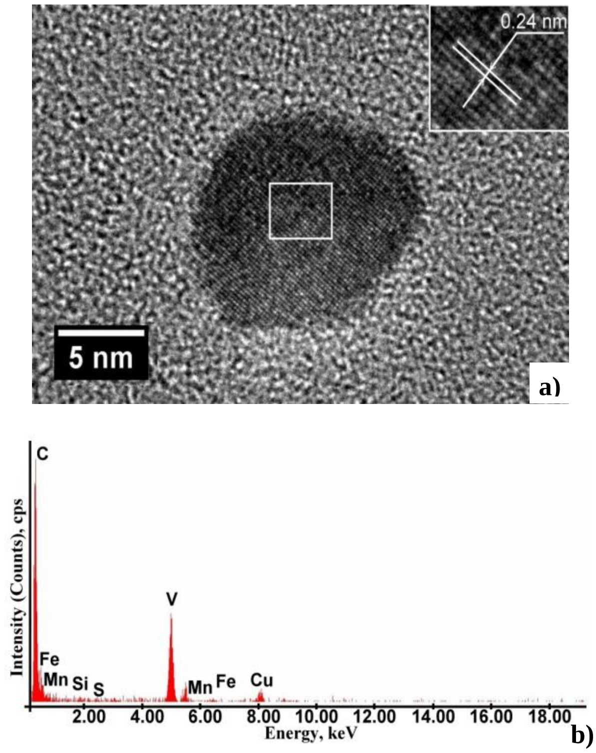

Fig. 2. (a) - The HRTEM micrograph and distance evaluation between the lattice fringes of VC nano particles and, (b) - EDX analysis of VC inhibitors

VC particles size is relatively fine with typical diameter from 5 to 15 nm and their dispersion is characterized by homogeneous distribution, see Fig. 2a. The result HRTEM and EDX analysis confirm that observable second phase particles are VC particles. The VC inhibitors are more

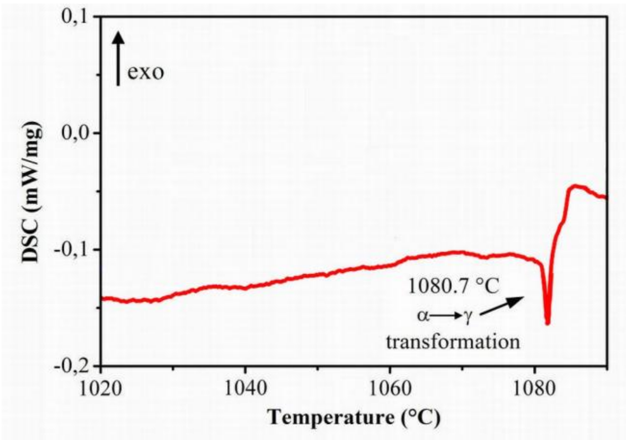

Fig. 3. The DSC measurements, which shows the presence α/γ phase transformation around 1080°C.

populated in the vicinity of grain boundaries but also uniformly distributed on the whole surface of carbon replicas. As one can see, the experimental sample is characterized by optimal distribution of VC particles and their size.

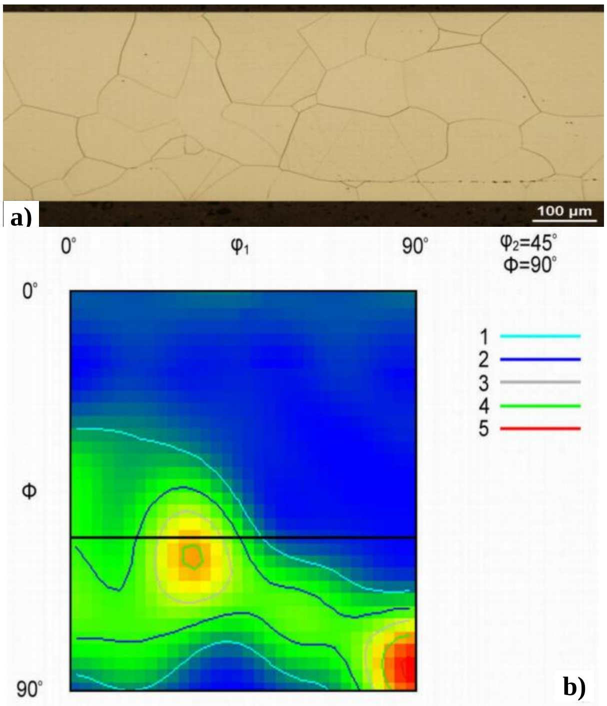

Fig. 4. (a) - the microstructure of experimental steel which presents the normal grain growth after annealing at 1075°C for 10 min, and (b) - ODF section taken at φ_2= 45° description the crystallographic orientation of grains which are presented in Fig.4a

Microstructural analysis of different grades of GO steels which were treated in our laboratory conditions clearly shows that optimal conditions for the abnormal grain growth are achieved approximately in the range temperatures from 1050°C to 1200°C. The value of temperature annealing is depending on the chemical composition of steels and kind of inhibitory particles.

On the other hand, it is well known that appropriate conditions for abnormal grain growth are observed close below the temperature of α/γ transformation. This temperature could be detected by differential scanning calorimetric analysis (DSC). The result of DSC analysis of experimental sample after decarburization annealing is presented in Fig. 3. The DSC signal was measured as a function of the heating temperature in the range from 1020°C to 1090°C at the heating rate 10°C/min. Despite the fact that the investigated steel belong to the vacuum degased steels with very low content carbon, the DSC measurements showed some endothermic effects which could be duo to the much higher concentration of carbon on the grain boundary in comparison with whole volume of material. It is possible that α/γ phase transformation could occur near the grain boundary. Over this temperature, the α and γ phases coexist and the ferrite grain growth is pined by the γ grains. On the other hand, at temperatures well below the α/γ phase transformation, the mobility of ferrite grains boundaries is markedly reduced [9]. Based on this data, the annealing temperature for our steel was chosen to be 1075ºC. This temperature is optimal for the grain growth of ferrite grains, see Fig. 5a.

Fig. 4a show the microstructure evolution of cold rolled experimental sample after the heat treatment at the 1075ºC for 10 minutes in the dynamic annealing conditions. Here, the microstructure of sample is characterized by the normal grain growth with average grain size ~200µm. As one can see this temperature of heat treatment keeps near the α/γ phase transformation region but is insufficient to start of abnormal grain growth. The abnormal grain growth is strongly pinned by VC second phase particles. It should be note that this microstructure was obtained after cold rolling without decarburization annealing. In order to achieve the abnormal grain growth for this experimental steel the additional driving force of grain growth was used. This

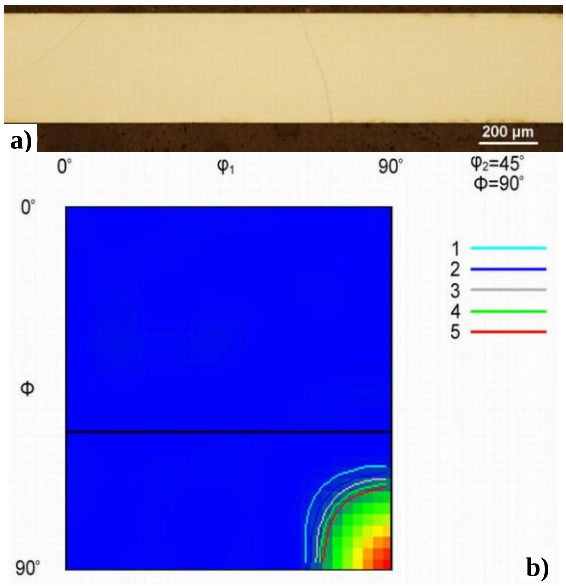

Fig. 5. (a) - the microstructure of experimental steel which was obtained after temper rolling with 4% of deformation and heat treatment at 1075°C for 5 min, and (b) - ODF section taken at φ_2= 45° description the crystallographic orien-tation of grains which are presented in Fig.5a

driving force was induced by means of the temper rolling process with 4% deformation. The microstructure evolution of the same material subjected to decarburization annealing with subsequent application of temper rolling with 4% of deformation and heat treatment at 1075ºC for 5 minutes in dynamic conditions is presented in Fig. 5a. As one can see the microstructure of this sample displays homogenous huge grains with average size of about one millimeter without other small parasitic grains or grains with secondary recrystallized matrix.

Figure 4b and 5b shows results obtained from the EBSD measurements which present intensities of particular crystallographic texture components. Here, EBSD analysis in the form of orientation distribution section (ODF) taken at φ_2 = 45° was carried out on the experimental specimen after the cold rolling and firs dynamic heat treatment and same samples after decarburization annealing, temper rolling and final annealing at 1075ºC/5min, see Fig. 4b and 5b respectively. As one can see, Fig. 4b shows that our laboratory steel annealed at 1075ºC/10min after cold rolling (see Fig. 4a) is characterized by deviation Goss texture (deviated from its ideal {110}<001> orientation) which is enhanced in comparison with so called deformed texture component {111}<110>. The domination Goss texture can be expressed the ε–fiber which shows marked intensity of the near Goss texture components (*ie* deviated Goss orientation). The γ fiber present intensity of deformed texture component {111}<110>, see Fig.4b. On the other hand the EBSD analysis show very strong Goss texture component of huge grains which was achieved after the complete abnormal grain growth with use the temper rolling process, see Fig. 5b. This fact suggests that abnormal grain growth with sharp Goss crystallographic orientation {110} <001> is achieved on the specimens which was treated by our proposed thermo mechanical method which is based on the use the combination of temper rolling process and subsequent final heat treatment in dynamic conditions. This method was applied to specimen with microstructural state presented in Fig. 5a.

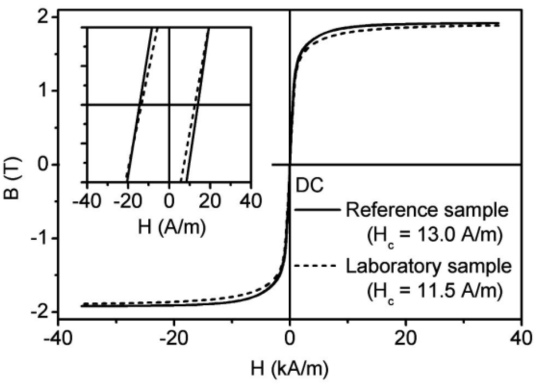

Fig. 6. DC hysteresis loops of the laboratory annealed sample and the reference industrial GO steel sample

The DC hysteresis loops of the laboratory annealed sample and the reference industrial GO steel sample are plotted in Fig. 6. The coercivity values were determined from the enlarged central part of the corresponding hysteresis loops. The achieved coercivity values of the laboratory treated samples are even slightly lower (DC conditions) to those of reference samples taken after industrial final box annealing. This means that the proposed laboratory dynamical heat treatment in combination with VC nano – particles can lead to the development of similar material's quality as the conventional process of GO steel production, however, at significantly reduced time.

The observed abnormal grain growth of steel with VC nano particles led to elaboration of sufficiently sharp {110}<001> Goss texture which is equal to that obtained in conventionally treated GO steels. Moreover, the steels treated by the newly developed method showed similar magnetic properties as the material which passed the conventional heat treatment (lasting tens of hours). This means that the proposed heat treatment and used VC precipitations at the appropriate coiling temperatures develops material of equal quality at significantly shortened time of heat treatment in comparison to the conventional rout of the GO steel production.

4 CONCLUSIONS

In summary we have shown that the observed strain induced abnormal grain growth in steel with the inhibitor system on the bases of VC nano - particles led to elaboration of sufficiently sharp {110}<001> Goss texture which is similar to that obtained in conventionally treated GO steels. Moreover, the steels treated by the newly developed method showed comparable magnetic properties to those observed for steels passed the conventional heat treatment (lasting several tens of hours).

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