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Effect of VC inhibitors in combination with unconventional dynamical heat treatment on the magnetic properties of GO steels.

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Abstract. The present work investigates some new approaches of grain – oriented steels processes. The suggested approach combines an application of nano – particles VC in combination with dynamic continuous annealing for secondary recrystallization in the investigated steels. Such a dynamical (fast heating) annealing and VC particles was applied to the grain – oriented steels in order to obtain abnormal grain growth with Gass crystallographic orientation development during secondary recrystallization. This abnormal gain growth led to evolution of suficienty sharp {110}<001> Goss texture which is equal to that obtained in conventionally treated GO steels. Moreover, the steels treated by the newly method showed similar magnetic properties as the materials passed the long – time heat treated. The coercive field value of our steels reached ~ 11 A/m. This means that the proposed heat treatment in combination with VC nano – particles lead to development equal material's quality at significantly shortened time of heat treatment in comparison to the conventional process of GO steel production.

1 Introduction

Grain oriented (GO) steels are iron 3% silicon alloys developed with a sharp {110}<001> 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 transformers of high efficiency [1]. The global movement in energy saving and environmental protection has aroused deep interest in the properties and total transformer performance of the GO silicon steel [2]. The industrial research activities are targeted on superior GO products with lower losses, higher permeability, and lower magnetostriction for the demands of more energy – efficient and less noisy transformers.

The strong Goss texture is a result of a technological route as far back as 1934 by Goss [3], which has been not significantly changed from those times. The procedure has been continuously improved and developed in order to achieve the best final properties of the materials [4]. Nowadays, the driving force for research and development are the increase of quality and reduction of the manufacturing costs. In the last years, these aspects have become the mainspring for most of the industrial research and development activities [5].

The Goss texture is evolved by a secondary recrystallization that takes place during the final box annealing that lasts several days. Despite the fact that the GO steels were invented quite long time age, the understanding of a scientific background of the Goss texture development and the exact mechanism of its formation in these steels is not fully understood. However, in order to achieving the {100}<001> texture development during the secondary recrystallization which is realized by means of the box annealing it is necessary to provide: (i) inhibitor of the normal grain growth by dispersion of small (50-100nm in size) second phase particle such as MnS, AlN and MnS+AlN, (ii) presence of the {100}<001> oriented grains in the primary recrystallized fine grained matrix [4]. In other hand the box annealing is a two step annealing process in which the first step is quasistatic heating where the grain – oriented steel is heated up to 1200ºC with a very low heating rate of 15 – 25ºC/h and the second one anneals at this temperature in around 30 – 40h.

Nevertheless, some new approaches have been discussed in the present work. The articles suggests completely new vision that consist in an application of nano – particles VC in combination with continuous annealing for the secondary recrystallization in the GO steels that would last several minutes. The goal of this work was to study the abnormal grain grows and texture development under the influence of VC particles, which were formed at lowest slab reheating temperatures, in grain oriented steel with new chemical content and subjected to unconventional heat treatment conditions. This proposed procedure will lead to a drastic reduction of production costs of the grain oriented electrical steels providing energy saving production with favourable impact on environment.

2 Experimental procedure

The material investigated in this work was a laboratory GO steels 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 smelting, the obtained slab which weighed 8 kg with 40mm thickness, was subjected to hot rolling, cold rolling and final annealing, see figure 1. A strip of 2,2 mm thickness was taken after final hot rolling process at 900°C and then these hot rolled strips of GO steels were subjected to three different coiling temperatures during 45 minutes, see figure 1. After this, strips of steel were subjected to cold rolled with reduction ε~84%, on the thickness 0.35 mm. and the decarburized process in wet atmosphere of H_2 (80%) + N_2 (20%) mixed gas (d.p. ~ +40ºC) This material was subjected to coiling temperature at 585ºC during 45 minutes in laboratory furnace. In this work, the influence of VC inhibitors on the magnetic properties of GO steel was investigated in materials which were holding at coiling temperatures 585°C and 650°C and was marked as C and E samples respectively.

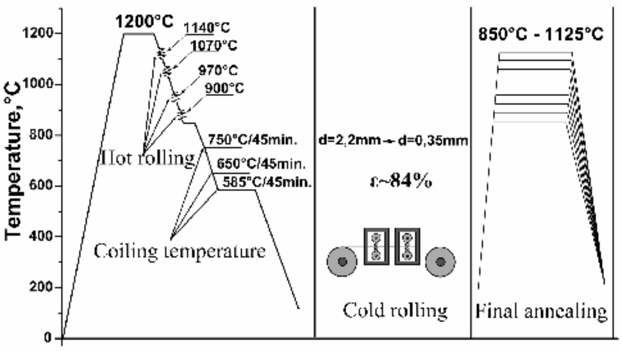

Fig. 1. Scheme of laboratory treatment of experimental materials.

The cold rolled samples were prepared by electric spark cutting into small strips with dimensions 3 cm × 1 cm with the longest side parallel to the rolling. A dynamic heat treatment was applied to these samples to study secondary recrystallization phenomena in the material. Each strip was heated to a different temperature in the range 850ºC – 1150ºC up to 10 minutes at a heating rate of more than 15ºC/s. The annealing time include the heating time, annealing time at selected temperature and cooling. The heat treatment was carried out in a dry hydrogen atmosphere.

Carbon extraction replicas for examination of VC particles in materials were prepared from metallographic samples and than were analyzed by means of JEOL 2001 transmission electron microscope (TEM) operated at 300kV.

After the annealing, coercive force measurements were performed on each sample. These measurements were carried out with an Oersted type coercive force meter KPC-1. Measurements of the full quasistatic hysteresis loops for the samples with lowest coercivity values (sample dimension 10cm × 0.5cm) were performed by using Forster type B-H loop tracer. The referent sample after a stress relief annealing under laboratory conditions at 800ºC for 60 min was also subjected to the measurements.

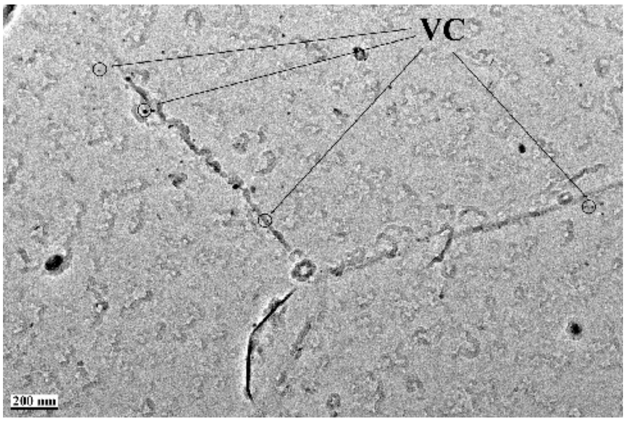

Fig. 2. Grain boundary and distribution of VC particles in material C.

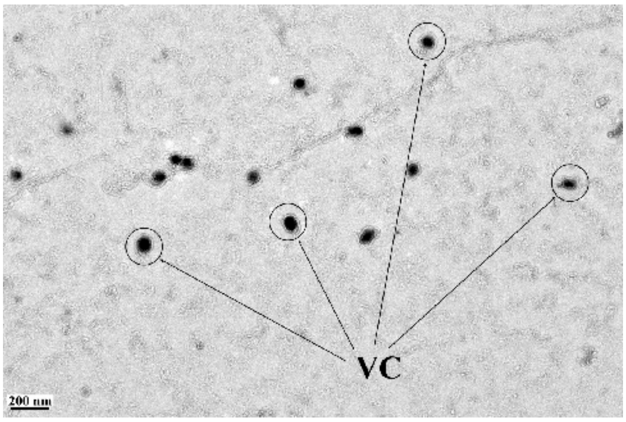

Fig. 3. Grain boundary and distribution of VC particles in material E.

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 for each sample of 2,5 cm x 1 cm in size. The JEOL JSM 7000F FEG scanning electron microscope was used to perform the texture analysis. The patterns of the back scattered electrons were detected by the "Nordlys-I" EBSD detector. The data obtained was processed by the CHANNEL-5, HKL software package.

3 Result and discussion

Observed dispersion of second phase VC particles in C and E materials after hot rolling process and different coiling temperatures are shown in figure2 and figure 3, respectively. The obtained fraction of the second phase particles in the mentioned materials are quite different. The figure 2 present the carbon extraction replica from C material after hot rolling process and coiling procedure at 585ºC during 45 minutes in laboratory furnace. As one

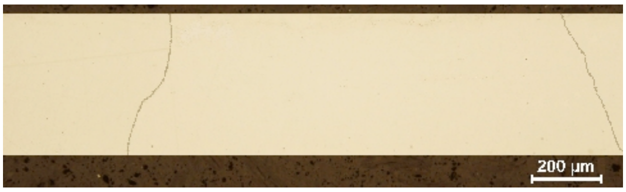

Fig. 4. The microstructure of the investigated GO steel C treated under the dynamical heat treatment conditions for 5 minutes at 1050ºC.

Fig. 5. The microstructure of the investigated GO steel E treated under the dynamical heat treatment conditions for 5 minutes at 1050ºC.

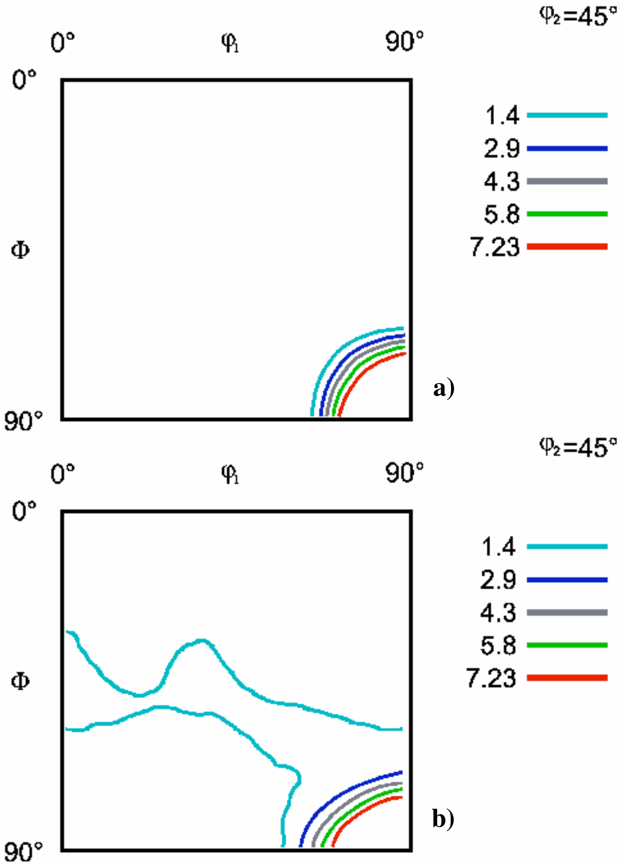

Fig. 6. ODF sections at ϕ_l=45° obtained from the investigated GO steels C (a) and E (b) treated under dynamical heat treatment conditions for 5 min at 1050°C.

can see, the VC particles mean sizes are in the range of 15 – 25 nm. The volume fraction of inclusions in this material is 0.1% and the most of the particles location near the grain boundaries. The figure 3 represent VC precipitations distribution in material E after hot rolling and coiling process at 650ºC during 45 minutes. The obtained fractions of the secondary phase particles in this

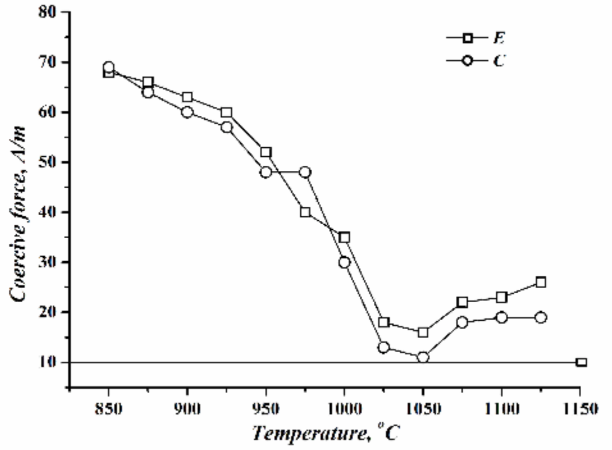

Fig. 7. Dependence of coercive force values on the annealing temperatures applied to the investigated C and E GO steels. Empty square corresponds to the Coercive force values measured on the GO steel taken after industrially box annealing.

material are approximately similar like in material C but the mean size of these particles is in the range 70 – 100nm. As one can conclude that the quantitative evolution of size and volume fractions of VC precipitations in materials C and E changes depending on the value of coiling temperature after hot rolling.

The abnormal grain growth (AGG) develops in the

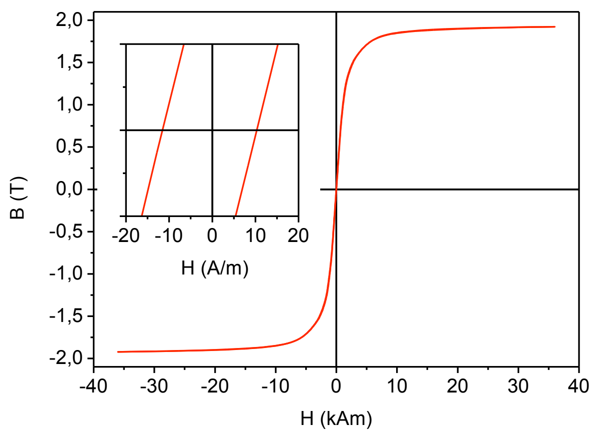

Fig. 8. Hysteresis loop of sample C after annealing at 1050ºC for 5 minutes in pure hydrogen atmosphere

samples C and E annealed after cold rolling and decarburization process at appropriate temperatures is presented in figures 4 and 5, respectively. As stated above, these materials were annealed at different temperature in the range 850ºC - 1125ºC. Further increase of the annealing temperatures up to 1000ºC leads to initiation of AGG in the present materials. The coarse–grained microstructure was obtained in both materials after annealing at 1025ºC in dry hydrogen atmosphere. It is important to note that the obtained microstructures of both samples differ by average grain size. The microstructure of C steel is presented in figure 4. This microstructure was achieved after annealing at 1050°C up to 10 minutes. The maximal average grain size of this microstructure is d = 1000µm, see figure 4. The microstructure of E sample after treatment at similar annealing conditions is presented in figure 5. The maximal value of average abnormal grain size of this material is d = 600 μm.

Figures 6a and b show the results obtained from the EBSD measurements which present intensities of particular crystallographic texture components. These measurements were obtained on the materials C and E after treated under dynamical heat treatment conditions up to 10 minutes at 1050ºC. Figure 6a present the ODF section at ϕ_1=45° obtained in steel C annealed at 1050°C up to 10 minutes. As one can see, this analysis show completeness of the abnormal grain growth with very strong Goss texture component. The ODF section at ϕ_1=45° obtained from the sample with such a microstructure as figure 5 shows a quite strong presence of the Goss texture and a weakened γ-fiber's texture components, see figure 6b. These facts suggests that abnormal grain growth of (110)[001] grains take place at this annealing temperature. However, these annealing conditions 1050°C/5min for material E are insufficient for the completeness of the abnormal grain growth of the Goss grains.

The measurements of coercivity were used to follow the changes in the microstructure and texture of the samples C and E. Figure 7 present change in the coercivity (H_C) as a function of the annealing temperature, for C and E samples. Value of the coercivity of untreated samples, after the final cold rolling, is about 700 A/m. This value drastically changes with applied annealing causing the primary recrystalization. As one can see the further increase of temperature results in minor changes of the coercive forces in C and E materials is in the range of 850º C - 950º C. The significant change of H_C in these steels is registered at 975ºC due to start of abnormal grain growth. The further decrease and plateau behavior of H_C curve in the temperature range of 1025ºC - 1075ºC should lead to the conclusion that the abnormal grain growth is completed in this temperature interval. Moreover, the coercive forces measurements for mentioned materials clearly show that their magnetic properties are not quite similar. The values of coercive field for C material are lower then for E. According to figure 7 the lowest coercivity value ~ 11A/m was achieved in C material after laboratory annealing at 1050ºC for 5 minutes. This low value of coercivity was confirmed also by the quasistatic hysteresis loop measurements as can be seen from Fig. 8. The value of H_C for the E sample after treatment in similar heating conditions was obtained near 16A/m. By the way, a comparison of the coercivity value of the laboratory treated sample with reference one shows a quite small difference between them.

The observed abnormal grain growth 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 passed the conventional heat treatment (lasting several tens of hours). This means that the proposed heat treatment and used VC precipitations at the appropriate coiling temperatures lead to development a same material's quality at significantly shortened time of heat treatment in comparison to the conventional rout of the GO steel production. This turns up in a drastic reduction of production costs of the grain oriented electrical steels providing energy saving production with favorable impact on environment. This proposed technological improvment is in a very good agreement with the main strategic policy of EU community.

Conclusion

A dynamical annealing of the laboratory grain oriented steel in combination of nano – particle VC was performed. There are two distinguished secondary recrystallization phenomena observed. The first is the abnormal grain growth of the Goss grains that was detected at relatively low temperatures 1025ºC – 1075°C under influence of unconventional dynamical heat treatment condition. The second one is the abnormal grain growth with very strong Goss texture component inhibited by VC second – phase particles in the range of 15 - 20 nm which were precipitated near the grain boundaries at the most lowest coiling temperature. In additions the achieved coercive forces values of the laboratory treatment samples are approximately similar with H_C value of references samples taken after industrial final box annealing. Even more remarkable improvement of the soft magnetic properties was observed for the C steel, where the coercivity value dropped to 11 A/m.

Acknowledgement

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