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RECRYSTALLIZATION OF NON-ORIENTED ELECTRICAL STEEL SHEET ALLOYED WITH TIN

RECRISTALIZACIJA NEORIENTIRANE ELEKTRO PLOČEVINE LEGIRANE S KOSITROM

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The orientation selective recrystallization of non-oriented electrical steels was studied. Microalloyed tin in electrical steels during recrystallization segregates to grain boundaries and on free surfaces. Tin segregation decreases the surface energy selectively, and the difference in the total energy of the grain is the driving force for its growth during recrystallization. This causes a selective effect on growth of differently oriented grains which finally leads to desired texture. The Auger electron spectroscopy (AES) was used to measure the grain boundary and surface segregation of Sn. The textures were measured by X-ray texture goniometer and the results were presented as orientation distribution functions (ODF). A transmission electron microscope was performed to observe the recrystallization processes.

Key words: non-oriented electrical steel, tin, surface segregation, grain boundary segregation, recrystallization, texture

Študirali smo, v odvisnosti od orientacije, selektivno rekristalizacijo neorientirane elektro pločevine. Mikrolegiran kositer med rekristalizacijo segregira na meje zrn in na proste površine. Segregacija kositra selektivno zmanjša površinsko energijo. Razlika v celotni energiji posameznih zrn predstavlja gonilno silo za rast posameznih zrn med rekristalizacijo. Različno orientirana zrna tako različno hitro rastejo, kar končno vodi do želene teksture. Površinsko segregacijo Sn in segregacijo po meja zrn smo merili s spektroskopijo Augerjevih elektronov (AES). Teksturo smo določili s teksturnim difraktometrom in rezultate prikazali v obliki orientacijskih porazdelitvenih funkcij (OPF). S transmisijskim elektronskim mikroskopom smo zasledovali rekristalizacijske procese.

Ključne besede: neorientirana elektro pločevina, kositer, površinska segregacija, segregacija po mejah zrn, rekristalizacija, tekstura

1 INTRODUCTION

Non-oriented electrical steel sheets are used as cores of electrical devices. Due to the fact that non-oriented silicon steel is mostly applied in small motors, its anisotropy should be assured. The ideal texture of non-oriented silicon steel would be a cubic fiber texture at which most of the grains have <001> axis parallel to the normal direction (ND) of the sheet.

It has been known that addition of small amount of Sn and some other tramp elements improves the magnetic properties of steels[1-5]. During the recrystallization process, Sn segregates at grain boundaries and on the surface. The Sn surface enrichment affects recrystallization by promoting the growth of grains with {100} plains parallel to the sheet surface. On the other hand, Sn grain boundary enrichment plays an important role in the secondary recrystallization[6].

The influence of Sn on the grain growth and on the texture development is shown in the present work.

2 EXPERIMENTAL

The experimental silicon steels were prepared in the laboratory. Vacuum melted and casted ingots were hot rolled, at a starting temperature of 1200 °C, to the final strip thickness of 6 mm and 2.5 mm. The strips were descaled and decarburized in a wet hydrogen at 840 °C. The silicon steels without and with 0.05% Sn of nearly the same following chemical composition: Fe, 2.0 % Si, 1.0% Al, 0.0015% C, 0.24% Mn. and 0.05% Sn, were obtained.

Segregation was studied "in situ" using Auger Electron Spectroscopy - AES. The Sn enrichment on the surface was determined by following the peak height ratio (PHR) of amplitudes between the dominant $Sn(M_5N_{45}N_{45})$ and the $Fe(L_3M_{23}M_{54})$ Auger transitions, located at the 430 and 651 eV kinetic electron energies, respectively. Both, surface and grain boundary enrichment of Sn were investigated by AES method.

The X-ray diffraction method was used for texture measurements. A goniometer using MoKα radiation was applied and the (200), (110) and (211) pole figures were performed. Additionally, orientation distribution functions (ODF) were calculated.

The recrystallization process was studied in the temperature range from 550 to 850 °C. Both steels with and without Sn were annealed in lead bath for different times from 1 to 60 minutes. The microstructure was examined by optical and transmission electron microscope. The average grain size was estimated. In addition an attempt was made to find the orientation dependence of recrystallized grains to the unrecrystallized.

3 RESULTS AND DISCUSSION

Sn added into experimental steels was in the range of solubility in α-Fe at all examined temperatures but it was

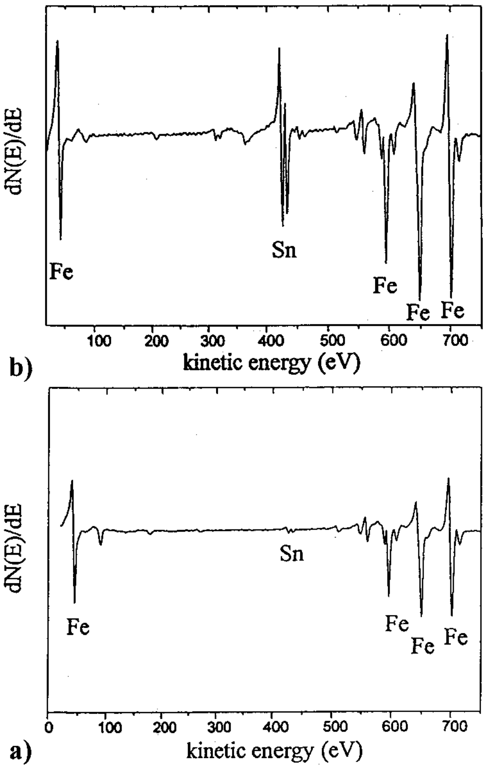

Figure 1. AES spectra of maximum equilibrium Sn segregation of steel alloyed with 0.05% Sn obtained **(a)** on the surface at 800 °C and **(b)** at the grain boundary aged at 550 °C for 200 hours.

Slika 1. Spekter Augerjevih elektronov maksimalne ravnotežne segregacije jekla legiranega z 0.05% Sn izmerjen **(a)** na površini pri 800 °C in **(b)** na mejah zrn po 200 urnem staranju pri 550 °C.

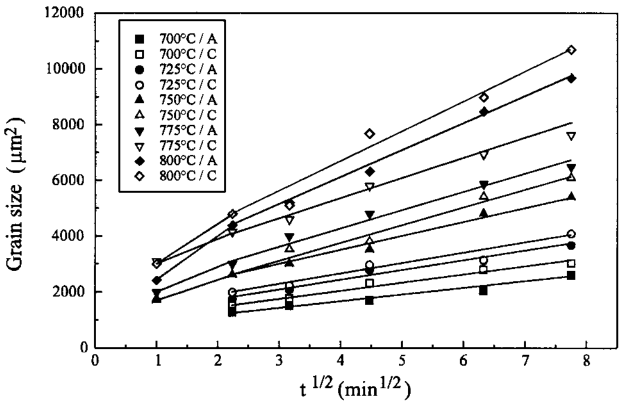

Figure 2. Grain size in dependence of annealing time for steel C with 0.05% Sn and steel A without Sn. Annealings were performed in lead bath.

Slika 2. Velikosti zrn v odvisnosti od časa žarjenja v svinčeni kopeli za jeklo C z 0.05% Sn in za jeklo A brez Sn.

below the detection limit of AES. After the specimens were exposed to higher temperature Sn enriched the surface, grain boundary and interfaces due to equilibrium segregation and its segregation was detectable by AES. All AES spectra were normalized to $Fe(L_3M_{23}M_{54})$ Auger transition at the 651 eV kinetic energy[7].

The highest Sn surface enrichment was established at 800 °C; above this temperature, no further increase in Sn concentration was observed (**figure 1a**). The equilibrium grain boundary segregation of Sn was attained after annealing the specimen for approximately 100 hours at 550°C (**figure 1b**).

The influence of Sn on the recrystallization was studied in steel with 0.05% Sn and in a comparison steel without Sn. The kinetics of grain growth and the final grain size were determined in the temperature range

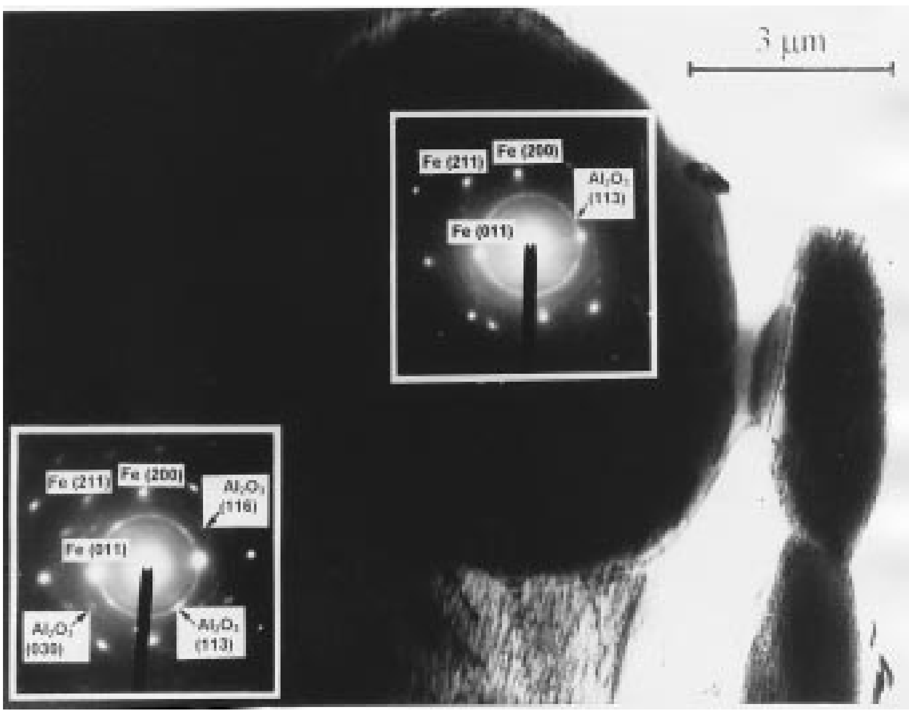

Figure 3. TEM micrograph of cold rolled non-oriented silicon steel sheet alloyed with 0.05% Sn, annealed 1 minute at 700°C with TED patterns of nucleus and unrecrystallized grain.

Slika 3. TEM posnetek hladno valjane neorientirane elektro pločevine z 0,05% Sn, žarjena 1 minuto pri 700°C in uklonska slika kali in osnove.

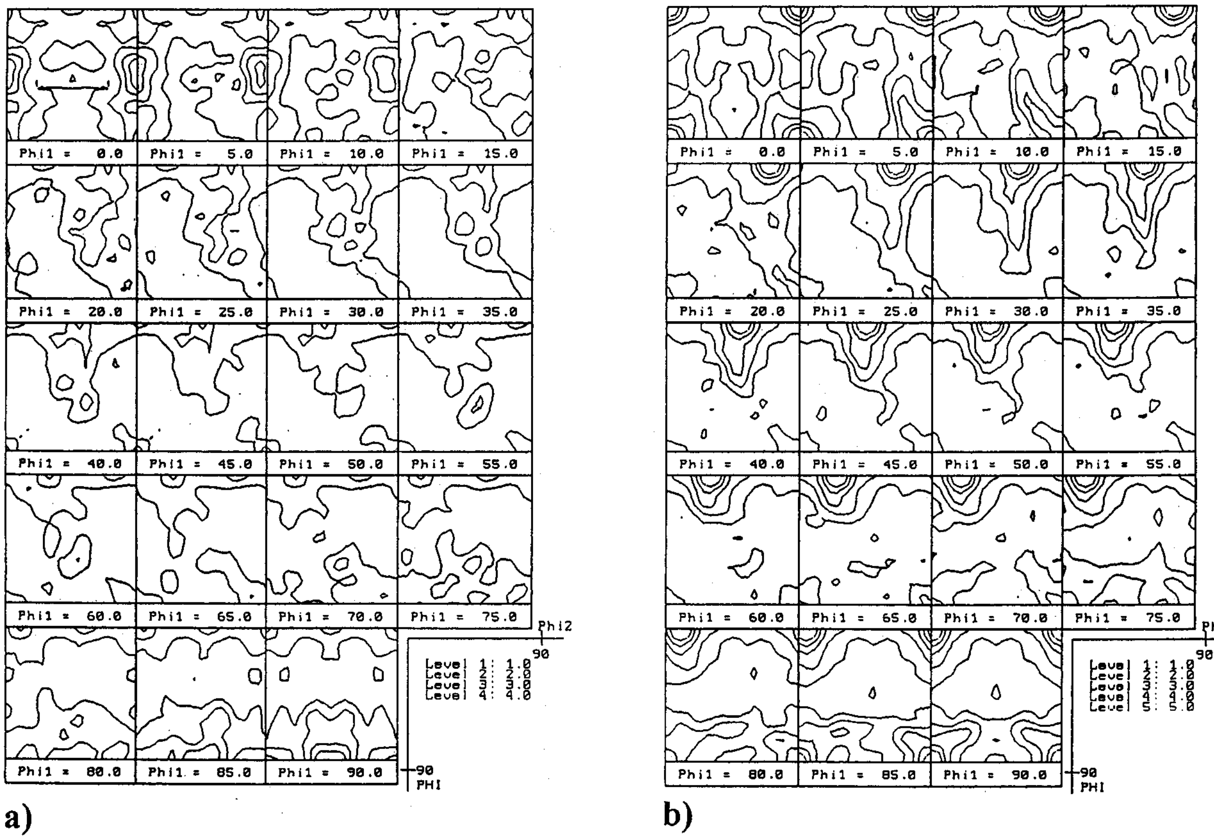

Figure 4. ODF recrysatllization textures measured on the surface of silicon steel **(a)** without Sn and **(b)** with 0.05% Sn.
Slika 4. Površinska rekristalizacijska tekstura silicijevega jekla **(a)** brez Sn in **(b)** z 0.05% Sn.

from 700 to 800 °C. Sn slightly suppresses primary recrystallization due to the solute drag effect[8]. There was no significant effect on grain growth rate, but it was found that the grains were coarser in the Sn recrystallized steel (**figure 2**).

The grain size in diameter of recrystallized grains annealed at 700 °C 1 minute was from 2.5 to 9 μm. **Figure 3** shows transmission electron micrograph of cold rolled silicon steel annealed at 700 °C 1 minute with transmission electron diffraction patterns of nucleus and unrecrystallized grain. Both diffraction patterns are in zone [011], so that the nucleus orientation to previous deformed grains is the same. Among Fe peaks one might notice the rings which belong to polycrystalline Al_2O_3 inclusions.

The textures of 0.5 mm thick electrical steels were measured on the surface in order to find out the influence of Sn surface segregation on texture development. The orientation distribution functions (ODF) f (g) were calculated from the (200), (110) and (211) pole figures. ODF function shown as a series of slices taken through the three dimensional ODF space is shown in **figure 4** and clearly demonstrates the difference in textures of steel without and with 0.05% Sn. The volume friction of grains having (100) planes parallel to the steel sheet surface increased in Sn steel for approximately once. The steels without and with 0,05% Sn have more pronounced {011}<100> and {001}<100> textures, respectively, while both steels have very low level of grains with plains (111), (211) and (411) lieing parallel to the steel sheet surface.

4 CONCLUSIONS

The grain boundary and surface segregation of Sn in non-oriented electrical steels with 0.05% Sn were determined. Maximum equilibrium segregation on the surface was reached at 800 °C. The equilibrium grain boundary segregation of Sn was attained after annealing the specimen for approximately 100 hours at 550 °C.

Sn surface segregation promotes growth of (100) oriented grains by selective diminution of surface energy. At the same time, Sn grain boundary segregation changes selectively the grain boundary mobility of certain grain boundary type.

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5 REFERENCES

1. S.Nakashima, K.Takashima, J.Harase, K.Takimoto, T.Kamijo: Mat. Sci. Forum 126-128 (1993), 419.

2. S.Nakashima, K.Takashima, J.Harase, K.Kuroki: J. Japan Inst. Metals, 55, 12 (1991), 1392.
3. K.Iwayama, K. Kuroki, Y.Yoshitomi, K.Homma, T.Wada: J. Appl. Phys., 55 (1984), 2134.
4. M.Jenko, F.Vodopivec, H.J.Grabke, H.Viefhaus, B.Praček, M.Lucas and M.Godec, Steel Research, 65, 11, (1994), 500.
5. M.Godec: Ph.D. Thesis, University of Ljubljana, Ljubljana 1997.
6. S.Suzuki, K.Kuroki, H.Kobayashi and N Takahashi: Mater. Trans., JIM, 33 (1992), 1068.
7. H.Viefhaus, M.Rüsenberg: Surface Science 159 (1985), 1.
8. J.Harase: Canadian Metallurgical Quarterly 34, 3 (1995) 185.