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Precipitation Reactions in a Copper – Bearing GOES

Vlastimil Vodárek[a]*, Anastasia Volodarskaja[a], Šárka Miklušová[b], Jan Holešinský[a], Ondřej Žáček[b]

[a]*VŠB – TU Ostrava, Faculty of Metallurgy and Materials Engineering, Ostrava, 708 33, Czech Republic*
[b]*ArcelorMittal Frýdek Místek, Frýdek Místek, 738 01, Czech Republic*

Abstract

The paper deals with minor phase evolution in a Cu – bearing grain oriented electrical steel during the following production steps of the AlN + Cu industrial processing route: hot rolling of slabs, the 1st cold rolling + decarburization annealing and the 2nd cold rolling + slow laboratory heating to the temperature of primary recrystalization. Thermocalc calculations were used for prediction of the effect of copper additions on equilibrium phases in the temperature interval 450 – 850 °C. Minor phase evolution was studied by using transmission electron microscopy. Hot rolling was accompanied by dissolution of copper rich sulfides. Slow cooling of coils after hot rolling resulted in precipitation of Fe_3C along ferrite grain boundaries. Decarburization annealing after the 1st cold rolling was accompanied by dissolution of Fe_3C, re-precipitation of fine Cu_2S, (Mn,Cu)S and very intensive precipitation of nitrides (AlN and Si_3N_4). Slow laboratory heating after the 2nd cold rolling to the onset of primary recrystallization (620°C) did not cause precipitation of any other minor phase. No copper rich metallic particles (ε - Cu) formed during the production steps investigated. Most fine particles of inhibition phases formed during decarburization annealing.

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1. Introduction

Magnetic properties of grain oriented electrical steels (GOES) depend strongly on the sharpness of the Goss texture ({110}<001>). It is believed that the perfection of the final texture is significantly affected by structural inheritance

* Corresponding author. Tel.: +00420 596 994 432 ; fax: +00420 596 994 401.
E-mail address: vlastimil.vodarek@vsb.cz

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during a complex processing route of GOES, Bernier et al. [1]. Factors which are considered to be very important for the formation of the Goss texture during high temperature annealing include the size of the initial grains with the Goss orientation, their orientation with respect to the other grains and the role of minor phases in grain boundary pinning [2]. Particles of inhibition phases (MnS or AlN, depending on a processing route) keep the ferrite grain size small during the early stages of the final high temperature annealing. Coarsening and dissolution of these particles makes the secondary recrystallization possible and the desired Goss-orientated grains grow and dominate the microstructure. It is believed that the final high temperature annealing is a critical factor in the processing, but there is still no generally accepted explanation. The industrial processing route of Cu – bearing GOES comprises of following production steps: slab reheating – hot rolling and coiling – the 1st cold rolling – decarburization annealing – the 2nd cold rolling – high temperature annealing – thermal flattening. According to Lobanov [3] copper could play several roles in GOES:

1. Increase the volume fraction and stability of austenite during hot rolling in the two - phase ($\alpha + \gamma$) region.
2. Small copper rich sulfides can inhibit grain growth during recrystallization processes (decarburization annealing and high temperature annealing).
3. Precipitation of ε - Cu could positively affect distribution of AlN, which are expected to be the most important inhibition phase [4]. Ideal conditions for precipitation of ε - Cu represents a slow heating rate during the initial stages of the high temperature annealing combined with the presence of a high density of lattice defects after the 2nd cold rolling. Zaveryukha [5] stated that precipitation of copper rich phase during primary recrystallization could retard precipitation of fine AlN particles to higher temperatures. Lobanov [4] speculates that a slow heating rate during primary recrystallization leads to precipitation of fine nitrides and copper segregations.
4. Segregation of copper atoms at grain boundaries can modify their mobility – dragging effect.

The aim of the paper is to characterize the effect of copper on precipitation reactions during basic steps of the industrially processing route of a Cu – bearing GOES.

2. Material and Experimental Procedure

The following production steps of the AlN + Cu industrial processing technology were studied: hot rolling of slabs (specimen A1), the 1st cold rolling + decarburization annealing (specimen A2) and the 2nd cold rolling + slow laboratory heating of sheets to the temperature of primary recrystallization (specimen A3). Chemical composition of the hot strip investigated is shown in Table 1. Hot rolling was carried out at 1250 °C to the thickness of 2.00 mm. After pickling, the 1st cold rolling to mid-thickness of 0.6 mm was applied and it was followed by decarburization annealing (DCA) at the temperature of 820 °C in the atmosphere containing N_2 + 20 % H_2. During DCA carbon content in the steel was reduced to 0.003 wt. %.

Table 1. Chemical composition of the hot strip, wt.%.

C	Mn	Si	S	Cr	Cu	$Al_{tot.}$	Ti	N
0.03	0.25	3.16	0.004	0.024	0.50	0.014	0.004	0.009

Subsequently the 2nd cold rolling was carried out to the final sheet thickness of 0.3 mm. Specimens cut from the industrially processed sheet were heated in a laboratory furnace to the primary recrystallization start temperature (620°C). The heating rate in a protective nitrogen atmosphere was v = 25 °C/h. Analysis of precipitation reactions was carried out using a transmission electron microscope JEM 2100 (TEM) for each step of processing. Electron diffraction and EDX techniques were applied for identification of minor phases on carbon extraction replicas. Electron backscattered diffraction (EBSD) in a scanning electron microscope Quanta FEG 450 was applied for evaluation of primary recrystallization during the slow heating of the cold rolled sheet.

3. Results and Discussion

3.1 Thermocalc simulation

The effect of copper additions to GOES on equilibrium phases in the temperature interval from 450 to 850 °C was simulated using the Thermocalc software. The TCFE 5 database was used. Chemical composition of the steel for