

STUDY OF THE INCLUSIONS FROM THE ORIENTED GRAINS SILICON STEELS AND THEIR INFLUENCE ON THE TEXTURE

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ABSTRACT

The paper presents experimental results concerning the inhibitor phases generation by microalloying elements utilization. It was made experiments on three silicon steel charges. It was observed that the inhibitor phases have a strong influence on steel sheet structure and its texture. So these phases can be considered like "texture nucleus".

KEYWORDS: inclusions, oriented grains, silicon steel, texture

1. Generalities

The extant scattered precipitates in the silicon steels have a decisive role, both: in grain forming, during secondary annealing and Goss texture evolution in the finished strip. The scattered precipitates appear in silicon steels as nitrides: NbN, AlN, as carbides NbC, TiC and sulphides: MnS, against to the added element in the steels to form growth "inhibitors" of the grainsize [1].

In the paper-framework it was watched the influence of the scatter-phase, formed by Niobium and Aluminum adding in the steel, on the evolution of the structure and Goss texture in silicon steels during secondary annealing. The experiments have been made on three steel grades with chemical composition shown in table 1.

Table 1. Chemical composition of experimental steels

Steel	State	Chemical composition %								
		C	Si	Mn	S	P	Al	Nb	O ₂	N ₂
N1	Casted	0.040	3.04	0.080	0.017	0.009	0.002	0.090	0.0073	0.0067
A2		0.040	3.02	0.070	0.023	0.013	0.070	-	0.0078	0.0065
M3		0.029	3.04	0.072	0.023	0.009	0.012	-	0.0076	0.0062
N1	Final plate	0.004	3.04	0.080	0.015	0.009	0.002	0.090	-	-
A2		0.004	3.02	0.070	0.020	0.013	0.070	-	-	-
M3		0.003	3.04	0.070	0.022	0.009	0.010	-	-	-

Steels N1 (fig.1.a) and A2 have been experimental made by manufacturing of two 50 kg ingots, then rolled and thermal treated in the Metal Rolling Laboratory from Galati University and M3 steel was industrial made by MITTAL STEEL S.A.

2. Experimental Researches

The test-specimen taken from those three steel-grades (hot and cold rolled strips) have been submitted to a complete investigations that watched:

- the nature of the scatter-phases and their role in the grain size growth process during primary

recrystallization (after first rolling) and secondary recrystallization (after second rolling)

- the role of the scatter-phase particles in forming and evolution of Goss type crystallographical texture

For study, the optical microscopy, electronically micro probe, X ray diffractometry and electronically microscopy have been used as investigation methods.

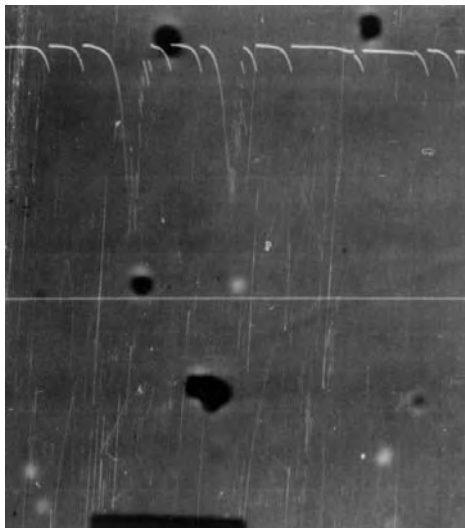
In that rolled strip, the optical microscopy distinguished the extant scatter-probe as rare and isolated small inclusions or rows of oxides, silicates and sulphides types.

A more complete analysis was made with electronically micro-probes specifying the nature and complexity of the scatter-phase particles by separation of the main elements in matrix and particles (fig. 1). In fig. 1, N1 steel appear about 1 μ m complex particle inclusions containing Mn, Al, Ca. Niobium appears isolated or with other elements, forming < 1 μ m diameter complex inclusions (fig 1a). In A2 steel are present: Al, Si, Mn (fig 1 d). Same type of the complex inclusions, containing Si, Al, Mn, appear in M3 steel. X-Ray diffractometry analyses distinguished crystalline structure of the secondary phase particles and matrix too. The fact of the analyses is that each phase characterized by its own crystalline net is corresponding to a specific system of diffraction lines. The test specimen have been taken from those three experimental steels, from hot rolled strips and submitted to a CoKa radiation. The results are centralized in table 2.

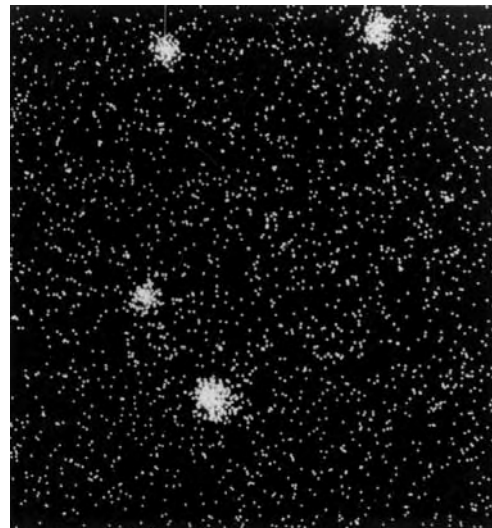
By this analyses, the evidence of the dispersion phase particles, with role of growth inhibitors of the grain size in silicon steel strip shave been watched.

In N1 steel, the microalloying with Niobium is determining the forming of the NbN and NbC precipitates with CFC type crystalline net.

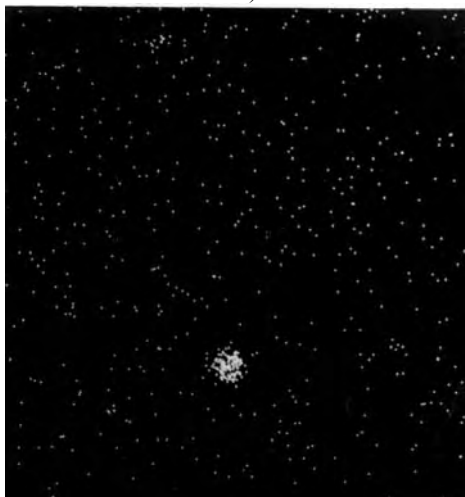
In A2 steel, with Aluminum, AlN Al₂O₃ are formed, both with hexagonal net, but in M3 steel is MnS present, with CFC type net. As literature of specialty shows (2) one of condition that should be carried out by the dispersion phase to be inhibitor is to create a non coherence degree between crystalline net of the particles and matrix. In case of the studied steels between ferrite matrix and dispersion phase particles: NbN, NbC, AlN, Mn, the complex relationship are established due to various crystalline net types and differences between values of the net parameters. These will determinate the net distortions, local elastic deformation and pressure exerted by the particles concentration on the grain borders, which will have as final effect the delaying, or even blocking the growth of the recrystallized grains [3]. Electronically microscopy analyses allowed for study the shape and size of the secondary phase particles in the finished strip.



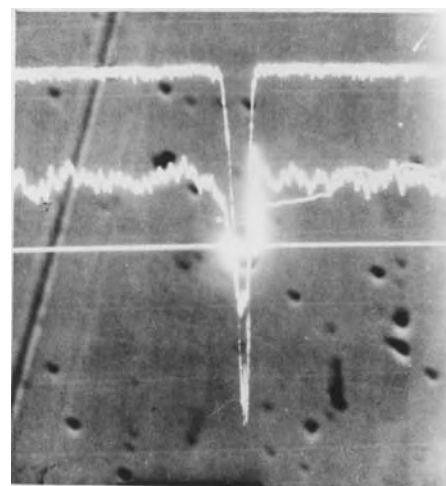
a)



b)



c)



d)

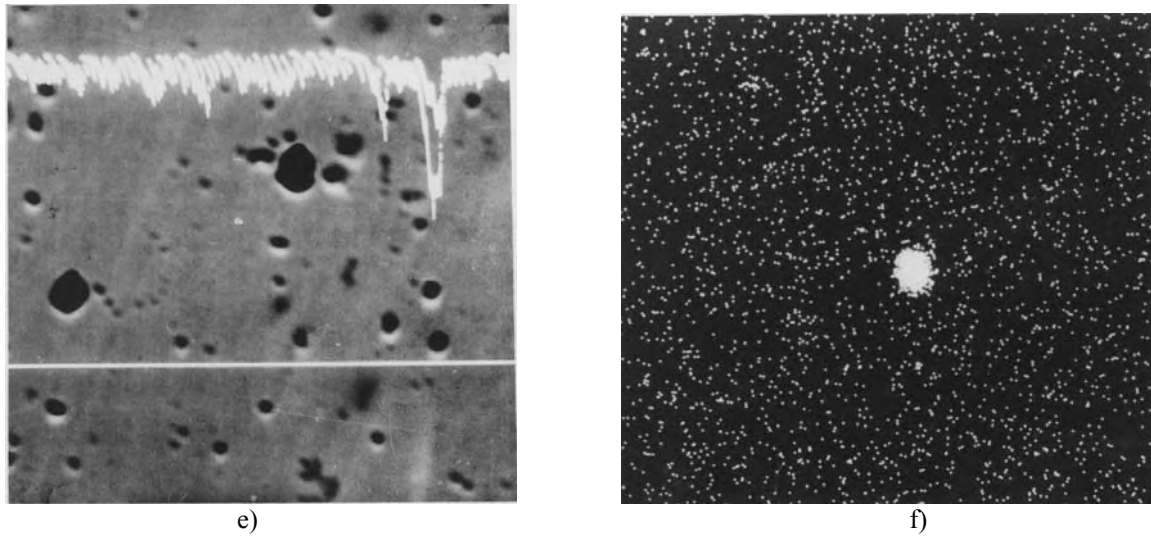


Fig 1. Complex inclusions in the experimented steels. a) Nb repartition in steel N1; b). Mn repartition in steel N1 c). Al repartition in steel N1; d) variation profile of Si and Al in steel A2; e). variation profile of Mn and Al; f). Al repartition in steel A2 (1200 x increase).

At temperature increase a suddenly increase of the grain might be produced either due to particle coalescence or their dissolving in matrix. For silicon steel, the distribution variance is wished to get a solid solution with a smallest quantity of inclusions, having in view the magnetic characteristics getting using thin foils method with transmission electronic microscope,

the test specimen of N1 steel was analyzed with 33,000 and 100,000x increase.

After secondary recrystallization it was established that NbN precipitates have left as small particle scattered in matrix of about 400Å, round or elongated shape (fig. 2).

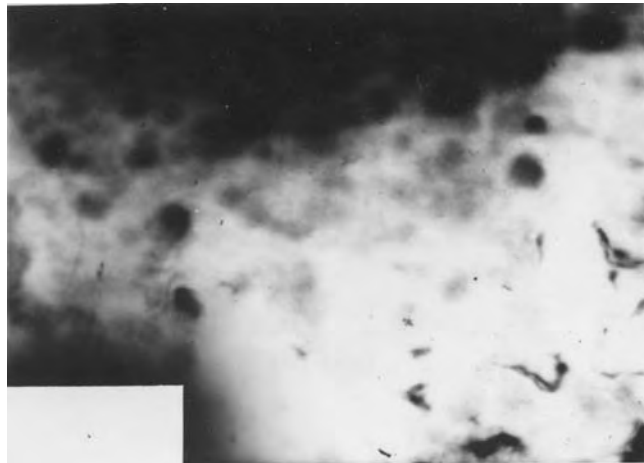


Fig. 2. Inhibitor particles in finished strip of N1 steel (33,000x increase)

Selection of a certain inhibitor should be correlated to the temperature of the secondary recrystallization too, to assure an advanced dissolution of the second phase particles, to get a certain grain and stressed Goss texture [4].

In this direction more ranges of secondary recrystallization have been experimented, analyzing, finally the percentage of the gotten Goss texture.

Strip test specimen of 0.35 mm thickness have been used, which have been thermal treated in Hydrogen atmosphere at 900°C, 1000°C, 1150°C temperature, 6 hours time. Fig. 3 shows the variation way of Goss texture component with secondary recrystallization temperature.

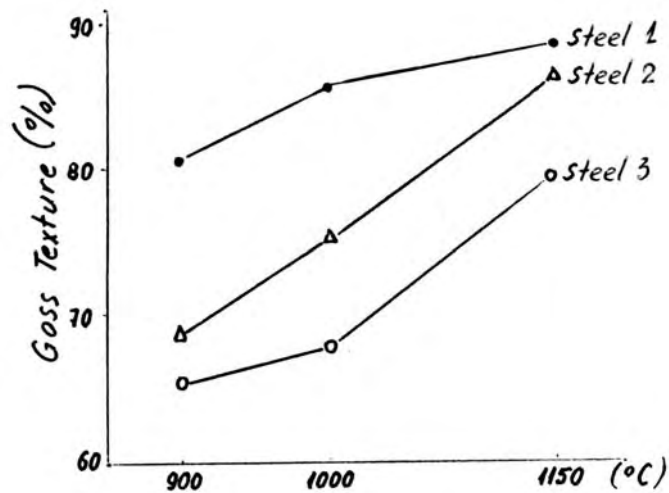


Fig. 3. Goss texture percentage to the annealing temperature for experimental steels.

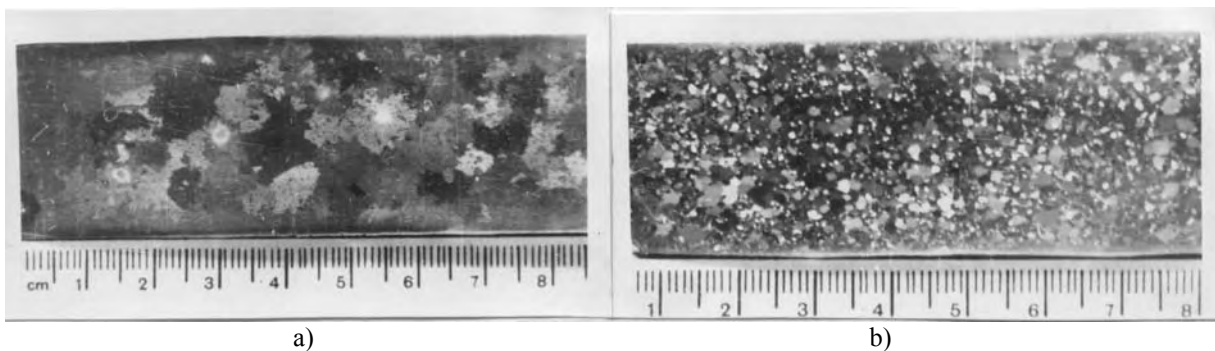
Could be remarked that from those three steel grades, this niobium steel answers the best to the requirements imposed to an inhibitor NbN is less soluble at lower temperatures (900-1000°C) than MnS, therefore, it can keep the grain at small size up to the high temperature. In this time, Goss texture nucleus could be formed, if deformation degree was high enough for (110)[100] texture to store high quantity of energy. At temperature over 1200°C, NbN particles are solving in higher quantity alloying grain growth, characteristic to the secondary recrystallization. At 1150°C, 6-8mm diameter grain size are formed in Nb steel. While in Mn steel (steel M3) very big grain size are forming with 15-20mm diameter. It is possible that, due to dissolution of a higher quantity of MnS particles, at lower temperature (850-900°C), equally, to produce the

growth of the other texture components in Goss texture detriment.

These suppositions have been confirmed by the texture measurements made on the experimental test-specimen: a percent of 24.8% texture (112)[100] resulted at 900°C temperature and 18% (112)[100] at 1000°C temperature. AlN particle have a stronger inhibition effect than NbN, dissolving in smaller quantity than NbN, at all temperatures.

As a result, we have remarked that over 1000°C the grains of Al steel (steel A2) remain small enough (under 5mm) and Goss texture is weaker.

In this case, the grain growth process is blocked by the presence of Al₂O₃ particles formed due to oxygen high quantity and very harmful by the negative influence on the magnetically characteristics of the finished strip. Fig 4 shows the macrostructure appearance after secondary annealing.



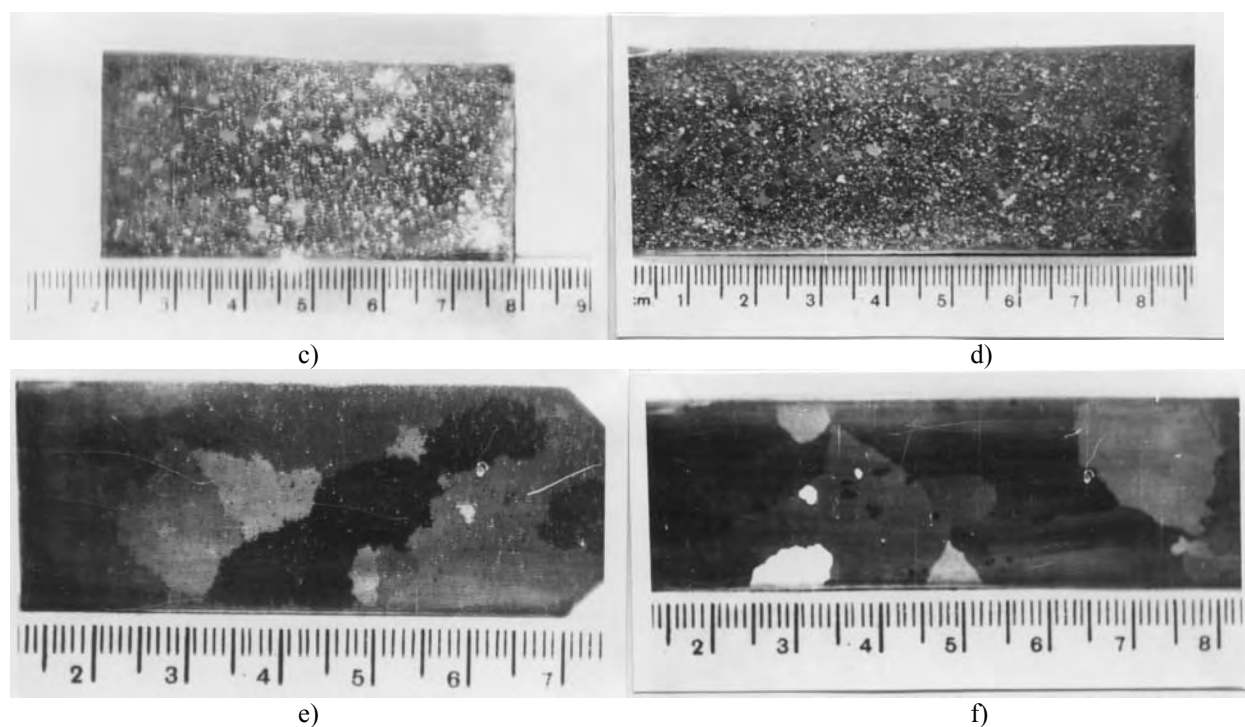


Fig. 4. Macrostructure of the strip after secondary annealing

a). steel N1 – annealing at 1150 °C, 6 hours, b). steel N1 - 1000 °C, 6 hours, c). steel A2 - 1150 °C, 6 hours, d). steel A2 - 1000 °C, 6 hours, e). steel M3 - 1150 °C, 6 hours, f). steel M3 - 1000 °C, 6 hours

Table 2. Crystalline nets characteristics of the dispersion phase – particles and matrix in the experimental steels.

Steel	Phase	2 θ (grades)	D/n	hkl	Crystalline net type	Crystalline net parameter
1	Fe ₃ C	44.08	2.38	(012)	Orthorhombic	-
	Ferită	52.74	2.02	(11)	CVC	2.98
	SiC	86.90	1.31	(201)	Hexagonal	-
	NbN	105.13	1.125	(400)	NaCl cubic type	1.10
	NbC	107.94	1.09	(400)	CFC	4.47
	NbC	130.00	0.993	(420)	CFC	4.47
2	AlN	44.38	2.37	(101)	Hexagonal	5.12
	Al ₂ O ₃	50.97	2.08	(102)	Hexagonal	-
	Ferită	54.00	2.02	(110)	CVC	2.98
	SiC	72.00	1.54	(110)	Hexagonal	-
	AlN	74.25	1.50	(233)	Hexagonal	5.12
	AlN	98.30	1.186	(202)	Hexagonal	5.12
3	Al ₂ O ₃	101.96	1.144	(131)	Hexagonal	-
	Ferită	53.20	2.02	(110)	CVC	2.98
	MnS	58.35	1.85	(220)	CFC	5.22
	Ferită	77.44	1.42	(200)	CVC	2.98
	MnS	89.26	1.12	(42)	CFC	5.22
	SiC	110.30	1.087	(203)	Hexagonal	-

From made researches resulted that inhibitor particles have an important influence on the texture [5]. It is possible that type of the crystalline net parameter, generally, non-coherence degree established between particles and matrix to influence, positively, the forming and growth of Goss texture nucleus. By the fact that in Nb steel is remarked the highest level of goss texture, it results that NbC or NbN inhibitor particles have the best influence on Goss texture. From here, could be concluded that the particles so called "inhibitors" of the grain size growth are, in fact "generator" of Goss texture too, acting as "texture nucleus" [6].

From those shown in this chapter results that the existence of the dispersion phases in silicon steels is a complex phenomenon with many implications on the structure and characteristics of the finished products.

Therefore, in the manufacturing process the following aspect, regarding the added elements for dispersion – phase – forming, should be taken into consideration:

- the accurate control of the chemical composition high purity and a very good steel deoxidation to stumple oxides formation which would limit phase forming with inhibitor role and texture nucleus;

- against to the technology used, chemical element (elements) choosing for dispersion phase forming;

- rigorous control of C and N contents, as dispersion phase to be, mainly, carbide and nitride, these being the most efficient;

- existence of the highest precision equipment to determinate nitrogen and oxygen contents from steel both in making process and finished strip as well.

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