



## Martensitic Transformations in High Alloy Fe-Ni Alloys

Stoyan PARSHOROV<sup>1</sup>, Evgeni MACHEV<sup>1</sup>, Petar PETROV<sup>2</sup>, Stefan VALKOV<sup>2</sup>

<sup>1</sup> Institute of Metal Science, Equipment and Technologies with Hydro- and Aerodynamics Centre  
“Acad. A. Balevski” at the Bulgarian Academy of Sciences, Sofia, Bulgaria  
e-mail: [s\\_parshorov@gmail.com](mailto:s_parshorov@gmail.com)

<sup>2</sup> Institute of Electronics at the Bulgarian Academy of Sciences, Sofia, Bulgaria

### Abstract

Three binary high-alloy Fe- 6, 12, 20% Ni with different stacking fault energy (SFE) were investigated. Based on the methods of internal friction and X-ray diffraction analysis, experimental results were obtained for the course and type of martensitic phase transitions during cooling. It is established that the structural features of the alloys determine the type of conversion mechanism. The role of SFE in the mechanisms of formation and growth of martensitic phases and the microstructural parameters of the solid solution in non-isothermal martensitic phase transformations is demonstrated.

**Keywords:** stacking fault energy, internal friction, martensitic phase transitions

## 1. Introduction

There are fundamental differences between the martensitic phase transitions in low- and high-alloy alloys. For the low-alloyed ones, when cooled at "supercritical" speed, they flow at a tremendous speed close to that of light, and the resulting martensitic phase is non-equilibrium and decays when heated. In these, the output austenite is characterized by high stacking fault energy (SFE) [1-3].

In high-alloy manganese and nickel alloys, the martensitic phase is equilibrium, does not decompose by heating, and their austenite is characterized by low SFE. Since SFE is related to the forces of atomic interaction in the crystal lattice, it is mainly determined by the type, amount of alloying elements in the austenitic matrix solid solution and temperature. Such alloys with “special” properties are of particular interest to the practice since they are the basis of a large number of stainless steels. The purpose of the present work is to investigate, by means of structural-sensitive methods, the peculiarities of phase transitions in the cooling of austenite in twin Fe-Ni alloys

## 2. Materials and methods

Object of investigation are three alloys Fe-5,8%Ni, Fe-12,4%Ni, Fe-20,6%Ni, produced in the IMSETHC-BAS. The specimens for investigation are manufactured by the following flow diagram:

- Casting of plates by vacuum furnace;
- The specimens are heat treated for finer structure – high temperature treatment – quenching from 1150°C after 10 min of holding – in water;

The present investigations are led using three structural-sensitive methods- X-ray diffraction – (specimens 15x15x5 mm), dilatometric – (specimens diam.5x50 mm) and the internal friction (specimens 50x1x1.2 mm) methods.

The X-ray structure analysis is performed by standard apparatus “Philips” using Co-K<sub>α</sub> ( $\lambda=1,7880 \cdot 10^{-10}$  m) emission. The speed and the step of the goniometer are as follows-

0,02°/min and 20 sec/step. Standard method for phase analysis of the martensite is used. The microstructure parameters of the martensite solid solutions – the domain size – D and the micro strains of the crystal lattice –  $\epsilon$ , are studied by the so called “single line analysis” shown in details in [4-7]. This method allows calculating the microstructure parameters of austenite and martensite on the basis of one diffraction line, for each of the phases.

The dilatometric tests were carried out with standard equipment "NETZSCH-Geratebau GmbH". Linear expansion values of standard samples in micrometers are recorded as a function of temperature and time. The tests were conducted in the temperature range 20 – 1200 °C with heating and cooling.

The apparatus for the internal friction measurements is built on the principle of the reverse balanced pendulum also known as Ke`'s pendulum. The operational frequency is in the range 1-1,3 Hz. The relaxation spectra of the specimens are recorded for the temperature range of 50-750K during heating and cooling in vacuum at rates of 10°C/min. The degree of the relative deformation during the measurements is  $5 \cdot 10^{-5}$  –  $5 \cdot 10^{-6}$ .

### 3. Results and discussion

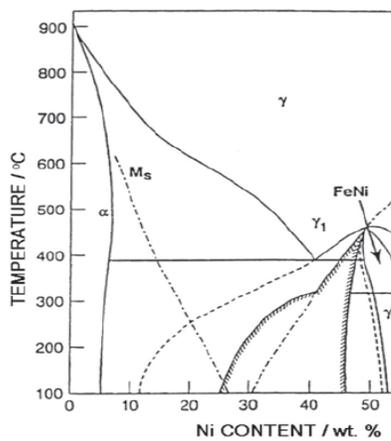


Fig. 1. State diagram – alloys – Fe-Ni [8]

According to the diagram of the state of Fe-Ni alloys, to a concentration of nickel of the order of 7-8%, under cooling austenite turns into ferrite and at higher concentrations into martensite – Fig. 1 [8]. The difference in the course of the two-phase transformations probably lies in the specific features of the microstructure of the initial austenite phase.

The general dependences of SFE on the concentration of alloying elements and temperature are shown in Fig. 2 and Fig.3.

The concentration dependence is observed at a minimum determined by the maximum solubility of the element in the defects in the arrangement, forming a Suzuki atmosphere, unlike the Cottrell atmosphere at full dislocations [2]. It can be seen that in the investigated alloys, as the concentration of nickel in the alloy increases, the SFE decreases – Fig. 2.

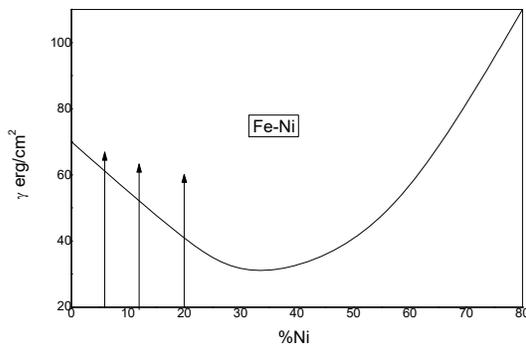


Fig. 2. Dependence of SFE on concentration of nickel in austenite

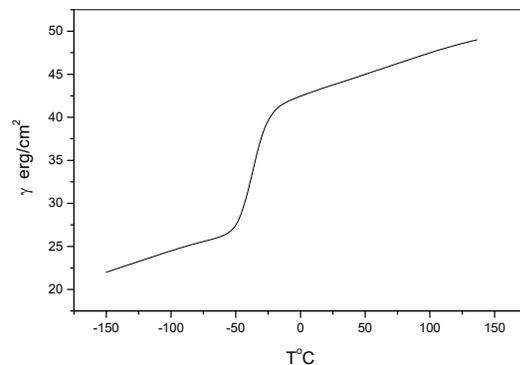


Fig. 3. Temperature dependence of SFE for the Fe-20% Ni alloy

Unlike Fe-Mn alloys with very low SFE, in which the non-diffusion phase conversion under cooling occurs with the formation of  $\epsilon$ -martensite [9], in Fe-Ni alloys the martensitic reaction is associated with the formation of  $\alpha$ -martensite.

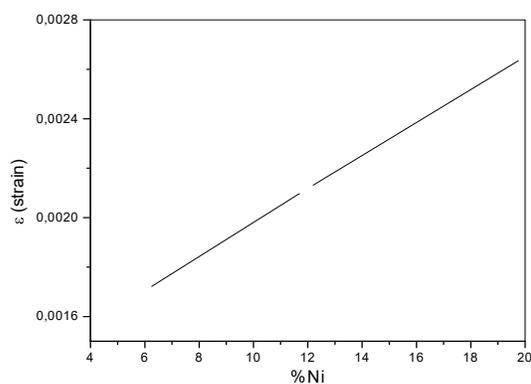
SFE is known to depend strongly on temperature, with the dependence being S-shaped, as shown in Figure 3, similar to phase conversion [2].

The results of the X-ray diffraction analysis of the investigated alloys showed that only the ferrite diffraction lines were observed in the 6% Ni alloy, and the presence of martensite and residual austenite in the order of 30% was observed in the alloys with 12% and 20% Ni – Table 1. This fact is related to the higher internal stresses in the interfacial spaces of the solid solution in the higher nickel alloyed alloys.

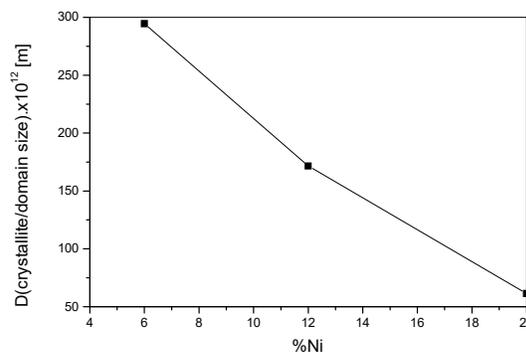
**Table 1. Crystal domain size  $D$ , lattice micro-deformation  $\epsilon$  and amount of residual austenite – in % by weight**

Sample №	Alloy-Phase	$D \times 10^{12}$ [m]	$\epsilon$	Austenite -%
1.	Fe- 20%Ni-(111) $\gamma$ austenite	61,385534	0,002303695	30,5%
	Fe- 20%Ni-(110) $\alpha$ martensite	76,97610176	0,002651967	-
2.	Fe- 12%Ni-(111) $\gamma$ austenite	171,5845778	0,002568146	31,7 %
	Fe- 12%Ni-(110) $\alpha$ martensite	186,8743651	0,002128976	-
3.	Fe- 6%Ni-(110) $\alpha$ ferit	294,5746254	0,001704634	-

Fig. 4 and 5 show the dependences of the domain size and the micro-deformation of the lattice of the solid solutions on the nickel content. It is interesting to note that as the nickel in the alloy increases, the length of the crystal domains decreases and the micro-stresses increase.



**Fig. 4. Dependence of micro strains on the nickel contents**



**Fig. 5. Dependence of domain size on the nickel contents**

The concentration limit of the transition of the austenitic phase to ferrite or martensitic was found to be lower than that reported in the literature. Martensitic phases are formed even at nickel concentrations of the order of 12% by weight. This is of particular importance in relation

to the possibility of creating new martensitic steel-based alloy based austenitostabilizing elements such as nickel.

The mechanism of the martensitic transformation in the alloys under consideration is defined in the most general aspect by the thermodynamic, crystallographic and other features of the alloys studied. According to the literature [2], the Fe-20% Ni alloy is characterized by an SFE of the order of 50 erg / cm<sup>2</sup>, which is clearly the value that guarantees a martensitic conversion of the type  $\alpha \Rightarrow \gamma$ .

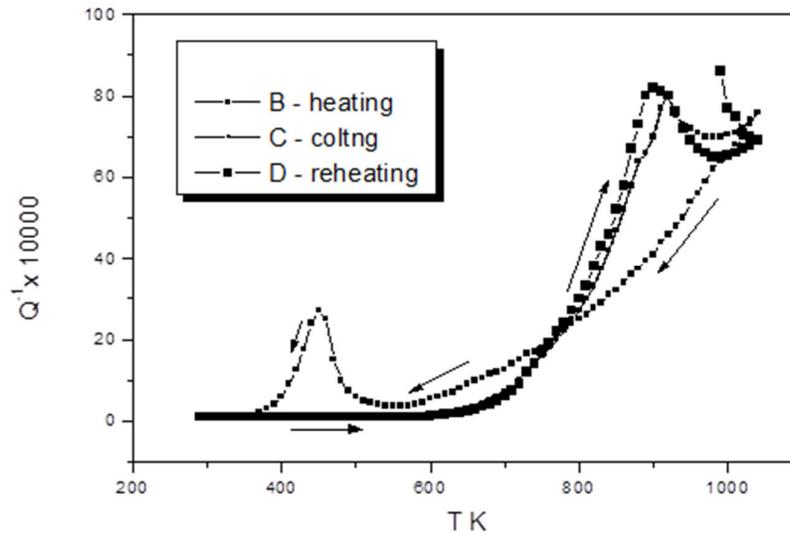


Fig.6. Relaxation spectra of alloy Fe-20%Ni

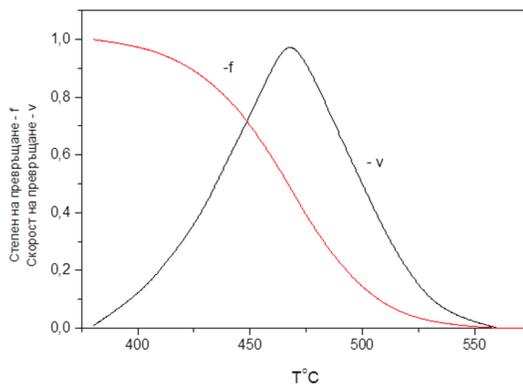


Fig. 7. Kinetics of  $\gamma \Rightarrow \alpha$ -martensite transformation obtained by integrating the relaxation maxima into an Fe-20% Ni alloy upon cooling of Fig.6

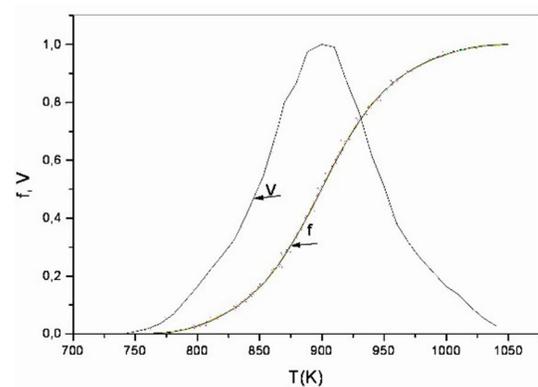


Fig.8. Kinetics of  $\alpha \Rightarrow \gamma$  transformation obtained by integrating the relaxation maxima into an Fe-20% Ni alloy upon heating of Fig.6

Based on published data in the literature, we have examined the relationship between the type of relaxation maximum and the mobility of the boundaries during phase conversion. Similar to our results for the martensitic reactions in Fe-Mn alloys [9], the symmetry of the relaxation maxima induced by the forward and backward reactions – Fig. 7 and 8, it was again found that these transformations were defined by the phase boundary movement of the new phase/martensite.

## 4. Conclusions

The results showed that:

- The concentration limit of the transition of the austenitic phase to ferrite or martensitic is lower than that reported in the literature. Martensitic phases are formed at nickel concentrations of the order of 12% by weight.
- The alloy Fe-6% Ni does not form residual austenite, which is associated with the flow of stress relaxation in the solid solution.
- Martensitic nickel alloys (12 and 20% Ni) have the same amount of residual austenite in the order of 30%, due to the high internal stresses in the interphase spaces of the solid solution.
- With the increase of nickel content and the formation of martensite, the microstresses of the grid increase.
- The symmetry of the obtained maxima and the lack of a degree of temperature dependence of the internal friction indicate that the relaxation processes in these alloys are defined by the phase boundary movement of the new phase / martensite.

**Acknowledgement:** The author is grateful to the financial support of Bulgarian National Science Fund at the Ministry of Education and Science, Contract No DN17/17 /12.12.2017/.

## References

1. <http://www.mtse.unt.edu/Banerjee/courses/Sp11PTlec13.pdf>
2. Petrov U., UF Journal, 16, 9,1971, p. 1409-1414.
3. Bhadeshia H. K. D. H., Martensitic Transformations: Crystallography and Nucleation, Encyclopedia of Materials Science: Science and Technology, eds K. Buschow, R. W. Cahn, M. C. Flemings, B. Iischer, E. J. Kramer and S. Mahajan, Pergamon Press, Elsevier Science, (ISBN 0-08-0431526), 2001, pp. 5203-5206.
4. Krumm S., WINFIT1.0 – A Computer Program for X-ray Diffraction Line Profile Analysis, XIIIth Conference on Clay Mineralogy and Petrology, Praha (1994), Acta Universitatis Carolinae Geologica 38(1994):253-261 Editum 24.1.1996.
5. Krumm S., WINDOWS-Software, <http://www.geol.uni-erlangen.de>.
6. Parshorov S., E.Marchev, Investigation of the unit cell parameters of Fe-Mn and Fe-Ni binary alloys, Congress MEEMI 2005, TU Varna, v. II, 160-163.
7. Machev E., S. Parshorov, Microstructure parameters of Fe- Ni and Fe- Mn alloys, Journal of materials science and technology, 2005, v.13, p.293-302.
8. Hansen M., Constitution of Binary alloys, Mc. 1958.
9. Parshorov St., P. Petrov, St. Valkov Martensitic Transformations in Mn Alloy with Low Stacking Fault Energy, Sixth European Conference on Crystal Growth, September 16–20, 2018, Riviera Holiday Club, Varna, Bulgaria, Under Review, <https://www.journals.elsevier.com/journal-of-crystal-growth>, <https://ees.elsevier.com/crys/default.asp>
10. Kelly P. M. The martensite transformation in steels with low stacking fault energy. – Acta met., 1965, 13, p. 635-646.
11. Benoit W., Thermodynamics of 2<sup>nd</sup> and 1<sup>st</sup> order phase transformations, Mechanical Spectroscopy with Applications to Materials Science, Q<sup>-1</sup> 2001 Ch.5.1, p.341-360.
12. Humbeeck J., The martensitic transformation, Mechanical Spectroscopy with Applications to Materials Science, 2001, Ch.5.3, p.382-415.