

EXPERIMENTAL RESEARCH REGARDING THE ROLLING TECHNOLOGY OF THE Fe-Ni ALLOYS

In the domain of the equipment and apparatus construction, a permanent preoccupation worldwide is ensuring technical performances and high fiability in exploitation. The users' requirement growth in this field led to producing materials with high characteristics such as iron-nickel alloys having a high nickel content with special magnetic, thermal, or elastic properties. The theoretical and experimental researches had the aim of obtaining cold rolled strip, thin (2.6 mm) and narrow (86 mm) from iron-nickel alloys with 41% Ni (low content of C: 0.02-0.04%; Fe: 58%; other elements: Mn, Si, Cu, Cr, Al: under 1%). Our own experiments aimed to establish an optimal cold rolling technology of hot rolled strips of iron-nickel alloys, in order to obtain cold rolled strips with superior mechanical and technological characteristics, strip profile according to current standards, including a finished product characterization.

Keyword: iron-nickel; rolling technology; mechanical characteristics

1. Introduction

Fe-Ni alloys are soft magnetic alloys endowed with high magnetic permeability that allows them to give an enormous magnetic response at small currents as required in electronics, telephony, automatics, computer technology [1].

In the experiments carried out in this paper we aimed to obtain thin and narrow strip, cold rolled from iron-nickel alloys with 41% Ni, which have physical, mechanical and technological properties corresponding to its use in the manufacture of measuring and control instruments, focusing on the following features:

- the material has a coefficient of thermal expansion as small as possible;
- minimal variations of the band thickness – both transversely and longitudinally;
- adequate appropriate surface, without microcracks, marks, etc;
- appropriate mechanical characteristics, in particular, a uniform hardness on the surface of the strip.

In the Fe-Ni system the phases α , γ , δ and γ' (Fig. 1) are formed. The α phase is a solid solution of nickel in Fe_α with c.v.c net. Because $\gamma \rightarrow \alpha$ transformation develops slowly, the position of the beginning and ending points of transformation A_3

is not precisely known than up to 773K; at this temperature the maximum solubility of the nickel in Fe_α is about 7% [2].

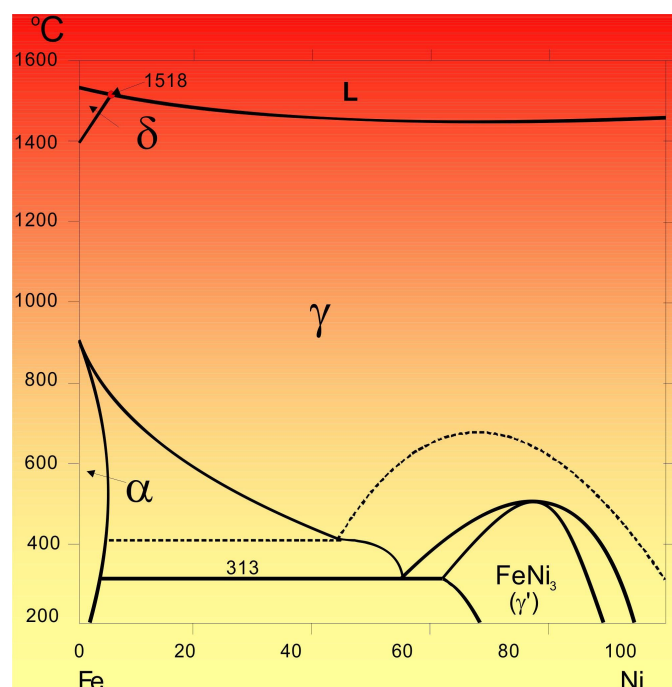


Fig. 1. The Fe-Ni phase diagram

¹ DUNAREA DE JOS UNIVERSITY OF GALATI, FACULTY OF ENGINEERING, MATERIALS AND ENVIRONMENTAL QUALITY RESEARCH CENTER (CMM), 47 DOMNEASCA STREET, RO-800008 GALATI, ROMANIA

* Corresponding author: mbordei@ugal.ro



The γ phase is a solid solution of nickel in Fe_γ , with c.f.c. structure. The δ phase is a solid solution of nickel in Fe_γ and the γ' phase, solid solution ordered on the $FeNi_3$, compound basis stable, under 773K [3]. Nickel strongly influences the coefficient of linear expansion of iron; the coefficient of expansion of iron-nickel alloys goes through a value close to zero at 36% Ni. By modifying the nickel content of iron-carbon alloys, steels with an expansion coefficient similar to certain materials can be obtained, so that they can be welded with the respective materials. Thus, steels with 40% Ni have the same coefficient of linear expansion with porcelain, steels with 0.2% C and 46% Ni have the same coefficient of linear expansion with platinum and glass [4-6]. Nickel has a particularly strong influence on the magnetic properties of iron. The initial magnetic permeability μ_a strongly increases to content of 78.5% Ni [7].

Cold plastic deformation reduces the magnetic permeability, but if it is followed by soft annealing, it leads to the formation of a crystalline texture that improves the magnetic permeability [8-10].

In order to obtain an optimal cold rolling technology, during the experiments, we used hot rolled strips made of an iron-nickel alloy with 41% Ni; the alloy has an austenitic structure, is ferromagnetic, and has a linear expansion coefficient with a value between 2 and $2 \times 10^{-6} C^{-1}$ at ambient temperature.

2. Experiment

The specificity of Fe-Ni alloys is given by the fact that, unlike other materials, in which the thermal coefficient of linear expansion increases with increasing temperature, at these alloys, at Ni content between 30-40%, there is an anomaly related to the non-variation effect of the coefficient, α , with temperature. Also, the normal temperature coefficient of the elasticity modulus, β , increases or remains constant with increasing temperature. The explanation for the abnormal behavior is of ferromagnetic nature: alloys of this type have a high volume magnetostriction, which is an increase in volume based on the inner magnetic field. Heating causes decrease of magnetostriction. Above the Curie temperature, the magnetostrictive deformations disappear completely, due to the fact that the metal passes into a paramagnetic state [11].

Due to the special characteristics, Fe-Ni alloys with Ni content between 30-40%, are used in the manufacture of some parts that require constant dimensions in variable thermal oper-

ating conditions: measuring and control instruments, geodetic devices, length standards, cryogenic technique. To ensure these minimum values for and stable construction dimensions, the carbon content must not exceed 0.05% [11].

The standard requirements for the studied material take into account the characteristics necessary to obtain the thin and narrow hot-rolled strip from Fe-Ni alloy, focusing on the following features:

- mechanical characteristics on the strip as uniform as possible;
- uniform hardness on the surface of the strip;
- coefficient of thermal expansion as small as possible;
- carbon content below 0.05%.

As reference material we chose: 42 Alloy, in conformity with ASTM F15 Alloy [12]:

- chemical composition: 41% Ni, 57.5% Fe, 0.05% C, other elements: Mn, Si, Cu, Cr, Al: 1.45%
- mechanical properties: (as annealed):
 - tensile strength: 565 MPa;
 - yield strength: 276 MPa;
 - HV10: 147.

For the experiments we chose an alloy with a content of approx. 58% Fe, 41% Ni, below 1% other alloying elements (Mn, Si, Cu, Cr, Al) and with a low carbon content (0.02-0.04%).

Some of the negative characteristics of the raw material used: oxide prints, thickness variations on the hot rolled strip width (approx. 4%), differences of hardness values on the length of the strip (up to 19.21 HV), have been eliminated through specific appropriate technological measures: pickling, heat treatment, etc.

The hot rolled strip coils were subjected to the pickling operation with sulfuric acid solution. The appearance of the surface was checked before and after the pickling process. The hot rolled oxide was removed by pickling process. After pickling, the coils were rolled on a quarto-reversible stand.

For chemical analysis, one sample was taken from each roll of hot rolled coil. To determine the chemical composition, a spectrometer of spark optical emission was used, specialized for the analysis of metal alloys, Magellan Q8 type. TABLE 1 shows the chemical compositions determined on the three hot rolled sheet rolls.

In this phase of experiments, there were used three coils of hot-rolled tape made of iron-nickel alloy with dimensions of 2.60×86 mm, aiming to achieve a cold-rolled strip of 0.5×82 mm.

TABLE 1

The chemical composition of the alloy used in experiments

Sample no.	The chemical composition [%]									
	C	Mn	Si	P	S	Cu	Cr	Ni	Al	Fe
1	0.03	0.25	0.30	0.002	0.002	0.07	0.13	41.2	0.16	Bal
2	0.02	0.21	0.32	0.002	0.002	0.06	0.12	41.0	0.16	Bal
3	0.02	0.22	0.32	0.002	0.003	0.06	0.13	41.0	0.15	Bal

Because during some previous experiments, at discounts of over 65%, there was a tendency to break the strip, a rolling program was carried out in two stages:

- **Stage 1:** the cold rolling coils were performed in four roll passes of reduction, from a thickness of 2.6 mm to a thickness of 1.1 mm (TABLE 2).

TABLE 2

Stage 1 of the cold rolling programme (four passes)

Pass no.	H [mm]	h [mm]	Δh/H [%]	
			Per pass	Total
1	2.80	2.20	21.4	21.4
2	2.20	1.60	27.0	42.0
3	1.60	1.25	21.8	55.5
4	1.25	1.10	12.0	60.0

- **Stage 2:** the rolled coils with the dimensions of 1.10 × 86 mm were cold rolled to a thickness of 0.50 mm, following the five-pass rolling scheme presented in TABLE 3.

TABLE 3

Stage 2 of the cold rolling programme (five passes)

Pass no.	H [mm]	h [mm]	Δh/H [%]	
			Per pass	Total
1	1.10	1.00	9.1	9.1
2	1.00	0.80	27.4	20.0
3	0.80	0.70	36.5	12.5
4	0.70	0.60	45.5	14.2
5	0.60	0.50	54.5	16.5

Between the two rolling stages, the strip was subjected to a soft annealing heat treatment inside the bell furnace according to the diagram represented in Figure 2. The coils were kept at a bearing temperature of 560°C for 120 minutes.

During the experiments we had in view and analyzed the evolution of the mechanical experiments and that of the structure.

From the samples taken from the hot and cold rolled strip, the specimens for the determination of the mechanical characteristics and the metallographic glyphs for the analysis of the microstructure were processed. The values of the mechanical characteristics presented in the tables after performing the mechanical tests represent the average of three tests for each coil.

The microstructural analysis was performed under an optical microscope by capturing the image using a video camera, the images being processed with a specialized program of automatic image analysis. Chromic acid (10 g) in 100 ml of distilled water was used for the chemical attack of the samples. The chemical

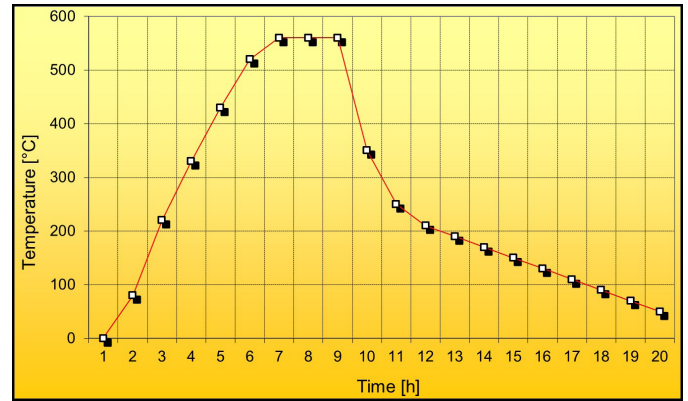


Fig. 2. Soft annealing heat treatment diagram for cold rolled strip

attack conditions were: electrolytic attack: 25 s; voltage: 1.5 V; distance between electrodes: 20 mm.

Several fields were investigated on all samples, and the recordings were made on the most characteristic areas.

3. Results and discussion

The mechanical characteristics of the cold rolled strip in the first rolling stage (corresponding to a total degree of reduction of 60%) before and after the heat treatment are presented in the TABLE 4 with its evolution highlighted in Figure 3.

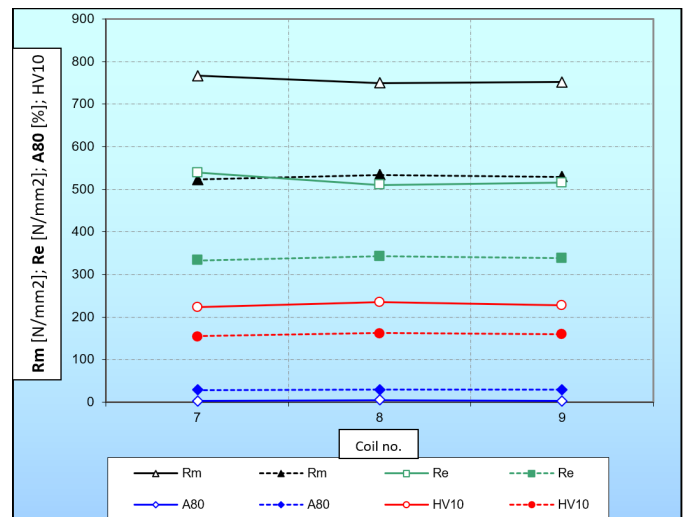


Fig. 3. The values of the mechanical characteristics on the cold rolled strip before (continuous line) and after thermal treatment of soft annealing (discontinue line) for 0.85 mm thickness

TABLE 4

The results of the mechanical tests for the samples, after Stage 1 cold rolling programme, before and after the heat treatment

Coil no.	Cold rolled strip				Cold rolled strip – thermal treated			
	Rm, [N/mm ²]	Re, [N/mm ²]	A80, [%]	HV10	Rm, [N/mm ²]	Re, [N/mm ²]	A80, [%]	HV10
7	767	539	3.8	223	524	333	29.2	155
8	749	510	3.9	235	534	343	29.6	162
9	753	516	3.8	228	529	338	29.4	159

The mechanical characteristics of the cold rolled strip in the second rolling stage (corresponding to a total degree of reduction of 54%) are presented in TABLE 5.

TABLE 5

The results of the mechanical tests for the samples, after Stage 2 cold rolling programme

Coil no.	Rm [N/mm ²]	Re [N/mm ²]	A80 [%]	HV 10
7	768	689	2.45	231
8	759	633	2.07	207
9	780	620	1.90	240

Analyzing the mechanical characteristics of the strip made of iron-nickel alloy of the 0.25 × 86 mm the following values should be taken into consideration:

- the tensile strength, R_m , had values of 750-783 N/mm²;
- the flow stress, R_e , had values of 658-674 N/mm²;

The microstructures obtained on the cold rolled strip are shown in Figures 4, 5 and 6.

The metallographic analysis of the produced strips emphasizes lightly hardened austenitic structure.

At a degree of cold deformation greater than 65%, without an intermediary treatment, the edges of the strips are subdued to the same extension stresses beyond the tensile load, provoking cracks or even marginal breaks.



Fig. 4. The hot strip rolling; thickness: 0.85 mm



Fig. 5. The hot strip rolling (thermal treated); thickness: 0.85 mm

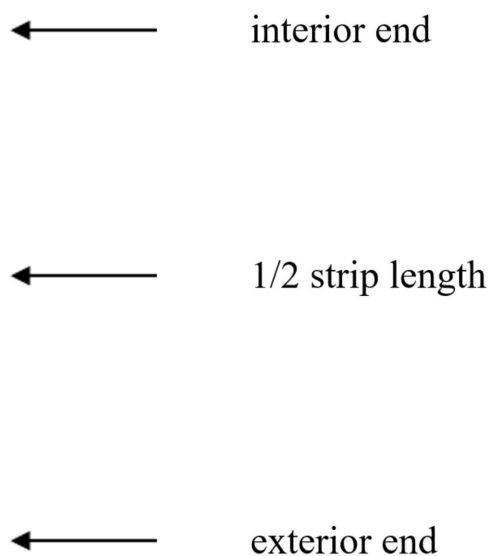


Fig. 6. Metallographic analysis of the hot strip rolling, thickness: 0.25 mm

4. Conclusions

Rolling is no longer a simple means of obtaining the final geometry of products in the metallurgical industry, because by leading in a certain way the rolling process it is possible to obtain a given structure, which might give the rolled products the desired mechanical characteristics. When choosing the technological parameters of rolling, the influence on these structural transformations that occur during deformation (recrystallization, phase transformation) will be taken into account.

The great ductility of the hot rolled strip allowed the cold rolling process, without the danger of breaking or cracking up to a hardening degree of maximum 65%.

To remove differences in hardness along the length of the hot-rolled strip, it is necessary to apply an annealing heat treatment before cold-rolling the thick strip.

The large differences in thickness in length, but especially in width, cause the corrugation on the cold rolled strip. The corrugations resulting from cold rolling due to variations of the laminated strip thickness, corroborated with the width of max. 2 mm of the cut out at the strips, determines the movement of the cold rolled strip during the striping and implicitly the non-observance of the width tolerances. It is necessary to ensure a hot rolled strip width of min. 88 mm for a final width of 82 mm, respecting the dimensions and thickness tolerances for the hot rolled strip.

The efficiency of using the metal for the cold rolled strip with the dimensions of 0.5×82 mm was 71.5%, the main losses being caused by the ends that cannot be laminated (21.03%). For hot rolled coils with an average weight of 100 kg, the length of the strip is approx. 55 m. Due to the reversible lamination that is practiced at the quarto stand, two ends of approx. 4 m at each coil, representing 14.5% for each intermediate rolling remain unrolled.

From the analysis of the variables that can influence the properties of the materials, it is possible to deduce the technological measures necessary to obtain the characteristics of the surface and the structure of the steels. Modern rolling mills allow the inclusion in tight tolerances and in addition to that the current rolling technique allows the exercise of some influences on the development of structures and on material properties. Modern rolling stands have high rolling forces and high-performance measuring and adjusting systems.

The variation of the mechanical properties on the width and length of the cold rolled strip imposes the determination of some improvement strategies of the equipment, for finishing the hot rolled strips, to obtain high quality in concordance with the consumer needs.

For this, it is important to use systems of modern management in order to satisfy the following requirements:

- an excellent dimensional precision of the thickness, both on the width and the length of the rolled strip;
- excellent flatness;
- uniform mechanical characteristics.

REFERENCES

- [1] V. Tsakiris, M. Petrescu, U.P.B. Sci. Bull. B **69** (2), 67-78 (2007).
- [2] K. Ito, Trans ISIJ (20), 624-628 (1980).
- [3] N. Geru, Structural theory of properties of metals (in Romanian), Didactic and Pedagogic Publ.House, Bucharest, 422-450 (1980).
- [4] M. Nili-Ahmadabadi, H. Shirazi, A. Fatehi, S. Hossein-Nedjad, Int J M P B **22** (18), 2814-2822 (2008). DOI: <https://doi.org/10.1142/S0217979208047638>
- [5] S. Qingshuang, H. Jun, Z. Xin, S. Zhonghua, Z. Yunfei, W. Yingfei, W. Z. Zhixiang, Z. Qiang, P. Huifen, Materials (Basel) **12** (8), 1297 (2019). doi: 10.3390/ma12081297
- [6] Z. Dong, W. Li, S. Schönecker, B. Jiang, L. Vitos, PNAS **118** (14), (2021). DOI: <https://doi.org/10.1073/pnas.2023181118>
- [7] M. Ludwig, PhD thesis, Magnetic properties of iron-nickel alloys under high pressure with relevance to planetary cores, Universität München, Germany (2014).
- [8] R.D. Enoch, A.D. Fudge, Br. J. Appl. Phys. **17** (5), 623 (1966).
- [9] B. Gehrman, J. Magn. Mater. **290-291** (2), 1419-1422 (2005).
- [10] W. Jamrozik, J. Górka, T. Kik, Materials **14** (2), 442 (2021). DOI: <https://doi.org/10.3390/ma14020442>
- [11] <https://vdocuments.net/materiale-compozite-aliaje-cu-memoria-formei.html>
- [12] <https://www.nealloys.com/pdf/nickel-iron-periodic-table.pdf>
- [13] Q. Sui, J. He, X. Zhang, Z. Sun, Y. Zhang, Y. Wu, Z. Zhu, Q. Zhang, H. Peng, Materials **12** (8), 1297 (2019). DOI: <https://doi.org/10.3390/ma12081297>
- [14] P. Alexandru, T. Radu, F. Potecasu, M. Vlad, G. Istrate, L.D. Buruiana, B.G. Carp, Rev. Chim-Bucharest **70** (4), 1466-1470 (2019). DOI: <https://doi.org/10.37358/RC.19.4.7150>
- [15] A. Arbogast, S. Roy, A. Nicz, M. Noakes, C. Masuo, S. Babu, Materials **13** (24), 5683 (2020). DOI: <https://doi.org/10.3390/ma13245683>
- [16] E. Bryk, G. Kang, S.C. Das Gupta, K. Mukherjee, TMS (The Metallurgical Society) (56); Conference: TMS-AIME fall meeting, Detroit, MI (USA), 16-20 (1984).
- [17] L.D. Buruiana, International Multidisciplinary 12th Scientific GeoConference SGEM 2012, Conference proceedings **4**, 685-692 (2012). DOI 10.5593/sgem2012
- [18] G. Cacciamani, J. De Keyzer, R. Ferro, U.E. Klotz, J. Lacaze, P. Wollants, Intermetallics **14** (10-11), 1312-1325 (2006).
- [19] W.S. Chang, Y. Wey, J.M. Guo, F.J. He, Open Journal of Metal **2**, 18-23, Published Online March 2012. (<http://www.SciRP.org/journal/ojmetal>)
- [20] D. Pieczaba, A. Jach, Arch. Metal. Mater. **55** (1), 25-35 (2010).
- [21] A. Romanski, T. Tokarski, D. Tyrała, Arch. Metal. Mater. **59** (3), 947-950 (2014).
- [22] A. Szabó, J. Kováč, A. Lovas, 27th International Colloquium of Advanced Manufacturing and Repair Technologies in Vehicle Industry, (5) (2011).
- [23] A. Szabo, R. Varga, F. Ujhelyi, V. Komanicky, A. Zorkovska, A. Lovas, Thermopower and Surface Magnetic Characterization of Ni Thin Layers, Acta Physica Polonica A **7** (1), 204-205 (2014).
- [24] G.O. Subramanian, C. Jang, J.H. Shin, C. Jeong, Materials **14** (1), 82 (2021). DOI: <https://doi.org/10.3390/ma14010082>