

Technical Sciences Academy of Romania <u>www.jesi.astr.ro</u>

Journal of Engineering Sciences and Innovation

Volume 2, Issue 3 / 2017, pp. 103-117

D. Chemical Engineering, Materials Science and Engineering, Natural Resources

Optimization of hot-metal working of austenitic stainless steels

IOAN ILCA*

University Politehnica Timisoara, Faculty of Engineering Hunedoara, Revoluției 5, Hunedoara

Abstract: The present paper ends the series of industrial research performed by the author with regard to the heating optimization of the austenitic stainless ingots. Austenitic stainless steels possess the best thermal shock resistance properties at low temperatures and the best resistance to elevated temperatures, with versatile demands in the economy.

For all stainless steel grades, the best hot workability is obtained when the structure of the steel at the hot-metal working temperature consists of a single phase. Small quantities of ferrite can be tolerated in the structure of the austenitic and martensitic steels as well as small quantities of austenite in the structure of ferritic steels, but these have to be maintained within certain limits, either through an appropriate change of the chemical composition of the steel, or through the control of the hot-metal working temperature.

Therefore, the research observes the evolution of the structure during the heating of austenitic stainless steels, stating the soaking zone favourable for reducing the existing content of ferrite down to values that do not worsen the plasticity.

Based on the conducted research, a new heating technology was designed, resulting in the reduction of the heating duration and a high efficiency in terms of the dissolution and spheroidization of the residual ferrite in the basic austenitic structure.

Keywords: stainless steel, austenitic structure, structural phases, thermal condition, phase composition, industrial ingots, deformability, good product (% quality product), batches.

1. Introduction

For several years, stainless steels consisted of iron and chromium alloys or iron, chromium and nickel alloys. Incidental, small quantities of certain elements were added, with the purpose of increasing corrosion resistance and improving their mechanical properties or in order to immunize steels against the action of harmful impurities. Recently, new stainless steel grades were developed, in which the

^{*}Correspondence address: ilca@fih.upt.ro

nickel content of the iron-chromium-nickel steels was entirely or partly replaced with combinations of nitrogen and manganese. The slow reaction velocities characteristic to stainless steels have prevented the determination of accurate balance diagrams; however, the existing diagrams to date allow at least the drawing of certain qualitative conclusions regarding the structure of these steels [1,2].

In terms of the corrosion resistance, the chromium content appears to be the determinant variable and the effect of the chromium can be enhanced by adding molybdenum, nickel and other elements [3]. The mechanical properties of stainless steels, much like those of other steels, are dependent upon the structure and composition of the material. Thus, the austenitic steels possess the best thermal shock resistance properties at low temperatures and the best resistance to elevated temperatures. Consequently, in that they exist in a great variety of structures, austenitic stainless steels display a wide range of mechanical properties which, combined with an excellent corrosion resistance, make these steels multilaterally used in the construction sector, transport equipment, sanitary equipment and cooking utensils, petroleum processing equipment, chemical products processing equipment, paper and food products equipment[4,5].

2. Content

Research into the determination of a heating regime for 3.6 tonnes ingots that would allow the dissolution of the secondary phases in the structure and the definition of the range with optimal plasticity was conducted at the Arcelor Mittal Company [6] and in the Materials Science Laboratories of the Hunedoara Faculty of Engineering.

At this company, the thermal condition for heating the austenitic stainless steel is determined depending on a series of factors, such as: the chemical composition, the phase composition, the heat distortion resistance, the type of furnace, the weight and sizes of ingots, the rolling diagram etc.

Given the fact that the structure of these ingots is not favourable from the point of view of deformability because of the extensive widening of the columnar crystal area and because of the strong segregation of the alloying elements which enable the appearance of constituents with a negative influence over plasticity, as well as a low thermal conductivity, it is recommended that the heating of the ingots up to approximately 800 °C be made at a low rate, while for higher temperatures it is possible to adopt higher heating rates. The optimal hot workability is obtained when the structure consists of a single phase [7,8,9].

Small quantities of ferrite can be tolerated in the structure of austenitic steels, but these must be maintained within certain limits, either by adjusting the processing temperature, or by the accelerated change of the chemical composition.

The segregated structure of the ingots is often a marked one, which entails the prolonged soaking at high temperatures in order to allow the homogenization of the structure and the diffusion of the segregation.

105

During heating, it is necessary to ensure an atmosphere low in sulphur, as sulphur can combine with nickel, resulting in nickel sulphide with a high tendency to break during working-up [10,11].

From the experience acquired to this date, we can mention that hot-metal working of austenitic stainless steels raises specific issues for each brand of steel and sometimes even for different batches of steel belonging to the same grade.

It is a well-known fact that the hot-deformability of a casted steel (in the form of ingots) is inferior to the deformability of the same steel plastically deformed beforehand.4

Not only that austenitic stainless steels make no exception from this rule, but the difference between the deformability of the two states is even more accentuated, as it can be seen in figure 1[12].



Fig. 1. Deformability of austenitic stainless steels in casted (a) and deformed state (b).

The chemical composition and an appropriate casting structure are prerequisites which influence the deformability of stainless steels, but these are not entirely controlled. Depending on the resulted chemical composition, as well as depending on other factors such as the sizes of the ingots, the casting and cooling conditions, the thermal heating conditions, the deformation process, emerges the presence of the second phase, namely ferrite, which leads to a sharp deterioration of plasticity[13].

A factor which worsens the steel's plasticity characteristics is the manner of conducting the melting process, more specifically the gas content and, in particular, the oxygen content. Thus, 0.01% O₂ leads to the deterioration and even to the rejection of the material.

For the manufacture of pipes from austenitic steels with molybdenum, highly stable, rich in nickel austenitic steels are recommended, such as 18-10; 18-12; 18-14 and not type 18-8 with Mo.

A 0.02 - 0.5% addition of cerium and lanthanum alloy is very conducive to these steels' deformability.

Some post-deformation defects may occur as a result of certain inclusions, especially of titanium nitride which attaches to the edges of the granules and results in scale-shaped defects.

Lead content in quantities of 0.01% in the composition of steel can lead to defects in the shape of tears during deformation.

The research conducted on a number of 70 batches of austenitic stainless steel lead to the conclusion that, provided the contents of Cr, Ni and Mo are ensured within the limits established by standards, the manganese content had a considerable influence on the deformability.

Thus, from a statistical processing represented in the diagram shown in figure 2, it resulted that, with a Mn removal, content of under 1%, entire batches were rejected. As the percentage of Mn increased, so did the percentage of good product, progressing to a point where the maximum quantity is obtained at a manganese content between 1.2 and 1.6%. The influence of manganese probably consisted of the contributory action of this element on austenitic stabilization.



Fig. 2. Influence of the Mn content on the deformability of austenitic stainless steels.

Even though Si is an alphagenic element, the results still showed that it needs to be present within certain limits, but no more than 0.50%, because its ferritizing effect can have a negative influence on plasticity.

Furthermore, the percentage of aluminium was limited to maximum 0.03%; taking into account the fact that it was not possible to ensure a content very low in non-metallic inclusions, especially oxides, it was necessary to limit the aluminium.

Once present in the structure, it should be possible to reduce the ferrite content by applying certain treatments for regenerating the austenitic structure, starting with prolonged soaking at temperatures of 1100 - 1200 °C, so that the ferrite matrix may dissolve or granulate.

The conducted research has shown that the experience acquired in this field in Romania matches the level of leading factories from abroad [14,15]. Thus, it has been observed that:

- Batches with a ferrite content of up to 5% have a good deformability. No important surface defects appear during their working-up;

- Batches with a ferrite content between 5 - 9% have a lower deformability. A more pronounced worsening of deformability can be seen at approx. 9% ferrite. Because of the formation of surface defects, in many cases the working-up of ingots must be interrupted at intermediate sections;

- Batches with a ferrite content over 9% have a very weak deformability. In most cases, their working-up must be interrupted because of marked surface defects. This leads to a low productivity, an increased energy, steel and manpower consumption.

At the same time, in order to achieve a good product procentage of approx. 70 - 72% finished products at these grades, only hot ingots are to be used (with a temperature of over 700 °C), taking into account the special precautionary measures that must be taken for heating cold ingots of these grades.

For this reason, the intended purpose of this research is to find a solution for dissolving the ferrite in the structural matrix of the steel and for improving the deformability, as well as for reducing the total heating-up period compared to the current technology.

For this purpose, a number of tests were conducted on samples taken from industrial batches, developed at Arcelor Mittal Hunedoara, at the end of last year.

Thus, four batches were taken into consideration, marked with 1, 2, 3 and 4, whose chemical composition is given in table 1.

Table 1

Batch	% C	% Mn	% Si	% S	% P	% Cr	% Ni	% Mo	% Ti
1	0.026	0.68	0.27	0.020	0.042	17.24	13.68	2.60	-
2	0.038	1.16	0.27	0.016	0.044	17.87	12.35	2.15	0.24
3	0.017	1.05	0.18	0.011	0.045	18.20	14.29	2.69	-
4	0.046	1.18	0.47	0.016	0.045	17.41	12.15	2.09	0.16

When analysing the ferrite content of batch 1, it was found that the industrially determined ferrite percentage in the workshop has fluctuated between 10 and 12%, and the investigated samples have confirmed these values.

For batch 2, the industrially determined ferrite content has fluctuated between 10 and 11%, and on the samples taken for research, it was between 8 and 10%.

For batch 3, the ferrite content did not exceed 8%, but on the investigated samples it fluctuated between 9 and 10%.

The industrially determined ferrite content for batch 4 was between 8 and 9%, and the samples taken have confirmed these values.

In order to study the dispersion of ferrite in the structural matrix, one of these ingots was cross-cut into sheets. It thus resulted that the ferrite dispersion, both in the same cross section and at various levels on the height of the ingot, is uneven. This is caused by the segregation of alloying elements in the mass of the ingot which takes place during the solidification and cooling down process.

The ferrite content formed during the solidification of the ingots and its distribution on the section depends both on the chemical composition and on the factors of the working - casting process (the casting temperature, the cooling down rate for solidification, especially in the range of elevated temperatures of over 1000 - 1100 $^{\circ}$ C).

The shortening of distances between ferrite islands and their elongation during the hot-metal working process leads to the acceleration of the diffusion processes. If the heating takes place in the ferrite minimum stability temperature range, then its solubilisation takes place. However, if the temperature is too high, the ferrite content might rise.

Analysing the topography of the points on the cross-sectional surface of the sheets obtained from cross-cutting the cast ingot, one can observe that the lowest ferrite content appears towards the exterior of the ingot. Towards its centre, the content increases continuously.

In general, the maximum level of ferrite in cross section is found at approximately a third of the side of the sheets, on both sides of the centre. When manufacturing pipes, for example, the area with the highest content of ferrite is precisely the area subject to the highest degree of deformation, the danger of breakage being, in this area, the highest. It is thus even more important to ensure a low ferrite content in order to improve hot-metal working and to increase the good product percentage.

Based on these preliminary findings, the actual research into the dissolution of ferrite and the shortening of the heating-up period was initiated.

To that effect, the research also used analytical calculations of the necessary heating-up time for the sheets resulted from the cross-cutting of the ingot.

Based on the analytical results of the determined heating-up times, as well as on the analysis of the current technology, different heating-up cyclograms were experimented, with the corresponding structural transformations.

Applying the heating-up regime given in diagram 1 in figure 3, the ferrite content of steel was reduced from 12% to 8%. The resulting structure emphasises the separation tendency of carbides in the area between granules, which represent tensional cracking fuses and which reduce corrosion resistance.



Fig. 3. Heating-up diagram 1



Fig. 4. Resulting microstructure after heating-up the samples according to diagram 1 Atac Nital 2%, 350 X

The occurrence of this phenomenon is caused by the insufficient cooling velocity. In order to avoid the carbides' separation tendency, several heating options involving the application of higher cooling velocities were experimented.

Thus, performing the heating according to the diagram shown figure 5, one can observe in the structure a slight spheroidization and granulation tendency of the ferrite, as well as a reduction of this phase from 12 to 8% (fig. 6).



Fig. 5. Heating-up diagram 2

Fig. 6. Resulting microstructure after heating-up the samples according to diagram 2 Atac Nital 2%, 350 X

Other experimented heating options (diagrams in figures 7 and 9) differ from the first ones by their complexity and the exposure time to various temperatures. The results obtained after the applied oscillating heating reveal a satisfactory reduction in the ferrite content, but in both cases, the form in which this constituent appears in the structure is undesirable, seeing that it encourages steel cracking during hot working.

The heating using this regime has also proven to be economically unprofitable given the high fuel consumption.

These reasons have determined the research of simpler solutions, easily reproducible in industrial conditions and with a more marked effect regarding the dissolution of ferrite.



Fig. 7. Heating-up diagram 3



Fig. 8. Resulting microstructure after heating-up the samples according to diagram 3 Atac Nital 2%, 350 X



Out of all these, diagram 5 shown in figure 11 is of interest to the industrial practice.

The analysis of the structure based on the heating under this regime (fig. 12) reveals favourable results in terms of the dissolution of ferrite from 12 to 5%, as well as a greater spheroidization tendency of the remaining phase.



Fig. 11. Heating-up diagram 5

Fig. 12. Resulting microstructure after heating-up the samples according to diagram 5, Atac Nital 2%, 350 X

The definition of the maximum range of hot-metal working temperature requires the investigation of phenomena occurring in the structure of the steel at higher temperatures. To this effect, the heating according to the diagrams shown in figures 13 and 15 was experimented, using samples with an initial ferrite content of 10%. When applying the heating according to the diagram shown in figure 13, the ferrite content rose to 16%, its settling down being visible in the structure (fig. 14) in the shape of nests, which is in fact ferrite δ , a constituent that worsens plasticity. The start of the range for ferrite δ occurrence is connected to the diagram shown in figure 15 and the respective structure which partially reveals a spheroidization of the ferrite, but, at the same time, a settling down tendency.



Fig. 13. Heating-up diagram 6



Fig. 15. Heating-up diagram 7



Fig. 17. Heating-up diagram 8



Fig. 14. Resulting microstructure after heating-up the samples according to diagram 6, Atac Nital 2%, 350 X



Fig. 16. Resulting microstructure after heating-up the samples according to diagram 7, Atac Nital 2%, 350 X



Fig. 18. Resulting microstructure after heating-up the samples according to diagram 8, Atac Nital 2%, 350 X

The trials conducted on samples for which the heating regime shown in diagram 8 in figure 17 was applied, have revealed a decrease in the ferrite content from 12 to 8%.

However, the analysis of the microstructure reveals a partial spheroidization of the ferrite, concomitant with a settling down of this phase into ferrite. (fig. 18).

An adequate study to reveal the influence of technological factors on the reduction of the ferrite content when working austenitic stainless steel would imply material costs and time, as well as the mobilization of productive capacities that cannot be made available to a research in operating conditions.

This is because only the cross-sectional and longitudinal cutting of industrial semifinished products could reveal the fluctuation of the structural secondary phases. Then, by applying various heating options, it would be possible to determine their efficiency in improving the structure.

For a more thorough knowledge of the phenomenon, the structural changes were investigated at elevated temperatures also using the "Vacutherm" microscope, using metallographic samples of the shape and sizes as the one in figure 19, taken from the centre of the experimental ingot.



Fig. 19. Metallographic sample for the "Vacutherm" microscope

It should be noted that vacuum heating facilitates the volatilisation of certain steel components. This phenomenon is more pronounced at temperatures of over 950 °C. The volatile components settle down on the protection plate of the microscope's lens and prevent the examination of the structure in the temperature range of interest.

Since the settling down took place quite rapidly, even after changing the plate, not all the photographs of the microstructures could be taken clearly.

Samples with different initial content of secondary phases (ferrite) were studied and selected, for which a soaking at temperatures of 1150, 1180 and 1200 °C was

applied. In addition to the structure, the reduction of the existing ferrite depending on the temperature and soaking period were also researched.

Figures 20 and 21 show clearer microstructures of some samples at different phases of the heating process. Thus, figure 20a shows the microstructure of the sample with an initial ferrite content of 13.5%. By maintaining this sample at a temperature of 1150 °C for an hour, the content of phase α in the structure dropped to 5%. Figure 20b shows the structure of this sample after a two hour soaking at a temperature of 1150 °C.

By increasing the temperature to 1180 °C, with a one hour soaking period at this temperature, fig. 20c, the ferrite content is maintained at 5% and we can observe a higher tendency of spheroidization of this phase in the base mass of the solid solution, a phenomenon favourable for the hot-metal working process. By continuing to raise the temperature to 1200 °C, with a one hour soaking period at this temperature, the ferrite content increased to 7.5%.

The microstructure of this sample is shown in figure 20d, resulting in austenite as a base constituent where the secondary phase, i.e. ferrite δ , is dispersed as elongated granules, an undesirable phenomenon, seeing that it is conducive to the creation of tensional fuses and a tendency for cracking, that is the appearance of cracks during the hot-metal working process.

Figure 21a shows the microstructure of the sample with an initial ferrite content of 9%. This microstructure shows several clusters of ferrite. After applying heat at 1150 °C, with a one hour soaking period, the microstructure shown in figure 21b results, which shows a rather homogenous dispersion of the ferrite and the absence of this phase's separation on the edges of the austenite granules.

By raising the temperature and by ensuring a one hour soaking period at 1180 °C, the ferrite content drops to 3%, being noticeable at the same time a more marked spheroidization tendency of this phase (fig. 21c). However, by increasing the temperature with just 20 °C and by maintaining the sample at 1200 °C for an hour (fig. 21 d), the quantity of ferrite increases to 4.8%.

This increase is of no particular importance for plasticity, but in the microstructure it is possible to see that this separation occurs on the edges of the austenite granules, which worsens the deformability of this steel.

Two important conclusion can be drawn from the results of the experiments:

- the most suitable temperature for reducing the content of secondary phases in the structure is between 1150 1200 °C.
- if this range is exceeded, the result is the separation of elongated ferrite δ, unfavourable for hot-metal working, as they represent tensional fuses for the formation of cracks.

114 Ioan Ilca/Optimization of hot-metal working of austenitic stainless steels



Fig. 20. The evolution of the structure and of the ferrite content at elevated temperatures for austenitic stainless steel with 13.5% ferrite

a – after a 1 h soaking period at 1150 °C; b – after a 2 h soaking period at 1150 °C; c – after a 1 h soaking period at 1180 °C; d – after a 1 h soaking period at 1200 °C. (Atac Nital 2%, 200 x)



Fig. 21. The evolution of the structure and of the ferrite content at elevated temperatures for austenitic stainless steel with 9% ferrite

a – after a 1 h soaking period at 1150 °C; b – after a 2 h soaking period at 1150 °C; c – after a 1 h soaking period at 1180 °C; d – after a 1 h soaking period at 1200 °C. (Atac Nital 2%, 200 x) In order to define the best heating regime, deformability tests using the hot-twisting method in the 1000 - 1250 °C range were carried out.

The obtained technological characteristics, namely the plasticity expressed by the number of twists until breakage (N) and resistance to deformation - expressed by the value of the moment of breakage (M), are given in figure 22.



Fig. 22. The technological characteristics resulted from the hot-twisting method.

It can be observed that the plasticity values are not high, however the maximum level is around the temperature of 1150 - 1200 °C. At the same time, a growth tendency of the ferrite content could be observed in the structure of the samples twisted at 1200 °C.

Linking the results of the deformability tests with the structural effects obtained from the oscillating heating tests, it is visible that the best heating temperature for working-up (the maximum level) is around the temperature of 1180 °C.

With these values, a new technology for heating the steel of this grade was developed (indicated with a full line in figure 23), compared to the current technology (indicated with a dotted line in the same figure), resulting in a reduction of the heating time and an increased efficiency in terms of ferrite dissolution and spheroidization in the basic structure.

The recommended temperature for the end phase of the working-up process is around 1000 $^{\circ}$ C, a claim based on the high values of the steel's resistance to deformation and on the lower value of plasticity caused by the high degree of alloying which results in a decrease in the recrystallization speed during the hotmetal working process.



Fig. 23. The optimized heating diagram for austenitic stainless steels for hot-metal working.

3. Conclusions

This paper is both a research and a practical tool for analysis and decision-making in the metal forming activity, for the purpose of ensuring the utilisation rate of the austenitic stainless steel finished product.

For an optimal deformability through hot-metal working, the structure of these steels must be monophasic, that is austenitic.

Seeing that the non-uniformity of the ingots' structure caused by segregation also generates areas with notable ferrite occurrence which worsen the plasticity, the research also investigated the influence of the dynamics of the heating process and soaking at elevated temperatures in order to reduce and dissolve the secondary phases in the base structure, below levels that could interfere with subsequent working through hot plastic deformation.

In order to highlight the evolution of the structure during heating, it was examined with the Vacutherm microscope and the best soaking area for reducing the ferrite content was indicated.

In order to guarantee the utilisation rate through a constant quality of semi-finished goods, metallographic research conducted at elevated temperatures, correlated with hot deformability tests, show the following conditions as advantageous and economic for the working up technology:

- the most suitable temperature for reducing the content of secondary phases in the structure is between 1150 1200 °C.
- if this range is exceeded, the result is the separation of elongated ferrite δ, unfavourable for plastic working, as they represent tensional fuses for triggering the formation of cracks and for reducing the good product percentage.

A new technology for heating austenitic stainless steels was developed, compared to the current technology (diagram shown in figure 23), resulting in a reduction of the heating time and an increased efficiency in terms of ferrite dissolution and spheroidization in the basic austenitic structure.

References

[1] William E. Luecke and John A. Slotwinski, *Mechanical Properties of Austenitic Stainless Steel Made by Additive Manufacturing*, Journal of Research of the National Institute of Standards and Technology, Volume 119, 2014.

[2] J. Bakajová, M. Dománková, P. Gogola, *Structural stability of high nitrogen austenitic stainless steels*, Materials Engineering - Materiálové inžinierstvo, **18**, 2011, p. 25-29

[3] Rajaković-Ognjanović and Grgur, *Corrosion of an austenite and ferrite stainless steel weld*, J. Serb. Chem. Soc., **76** (7), 2011, p. 1027–1035.

[4] Plaut et al., A Short Review on Wrought Austenitic Stainless Steels at High Temperatures: Processing, Microstructure, Properties and Performance, Materials Research, Vol. 10, No. 4, 2007, p. 453-460.

[5] B. Rouxel et al., Influence of the austenitic stainless steel microstructure on the void swelling under ion irradiation, EPJ Nuclear Sci. Technol., 2, 30, 2016.

[6] Samoilă C., Ionescu M., Drugă L., *Tehnologii și utilaje moderne de încălzire în metalurgie*, Editura Tehnică, București, 1986.

[7] Janovec et al., *Phases in austenitic stainless steels*, Materiali in tehnologije, 37, p.6, 2003.

[8] Bhadeshia N.K.D.H., Honeycombe R., *Steel Microstructure and Properties*, Edition III, Ed.Butterworth – Elsevier Ltd., USA, 2006.

[9] Z. Yanushkevich, A. Lugovskaya, A. Belyakov, R. Kaibyshev, *Deformation microstructures and tensile properties of an austenitic stainless steel subjected to multiple warm rolling*, Materials Science & Engineering A 667, 2016, p. 279–285.

[10] Jun-Xia Huang, Xiao-ning Ye, Zhou Xu, *Effect of Cold Rolling on Microstructure and Mechanical Properties of AISI 301LN Metastable Austenitic Stainless Steels*, Journal of Iron and Steel Research, International ,Volume 19, Issue 10, p. 59-63, 2012.

[11] Guixun Sun, Yu Zhang, Shicheng Sun, Jiangjiang Hu, Zhonghao Jiang, Changtao Ji, Jianshe Lian, *Plastic flow behavior and its relationship to tensile mechanical properties of high nitrogen nickel-free austenitic stainless steel, Materials Science and Engineering: A*, Volume 662, 2016, p. 432–442.

[12] Rațiu S., Alexa V., Ilca I., *Cercetarea deformabilității oțelurilor inoxidabile termostabile*, Conferința Națională cu participare Internațională, Ediția a IV-a, Septembrie 2016, Cugir, România, p. 109.

[13] Ilca I., Magaon J., *Structural research on semi-industrial high temperature stainless steel, thermostable*, VII International Congress, "Machinery, Technology, Materials" Sofia, Bulgaria, 2010.

[14] Ilca I., *The theory and practice of heating quality steel ingots*, Journal of Engineering Science Innovation, Volume 1, Issue 1, 2016, p. 120-130.

[15] Ilca I., *The determination of the gradient and thermal stresses for the heating of type 18-10 stainless steel ingots*, Journal of Engineering Science Innovation, Volume 2, Issue 2, 2017, p. 58-74.