ODREĐIVANJE DELTA FERITA U AUSTENITNIM NEHRĐAJUĆIM ČELICIMA

DETERMINATION OF DELTA FERRITE IN AUSTENITIC STAINLESS STEELS

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REZIME

Mikrostruktura austenitnih nehrđajućih čelika je uglavnom monofazna, tj. austenitna. Međutim, hemijski sastav čelika može utjecati na izdvajanje delta ferita. Prisustvo delta ferita u austenitnom nehrđajućem čeliku ima koristan ili štetan utjecaj na mehanička svojstva (udarna i zatezna), kao i na zavarljivost i otpornost na koroziju. U ovom radu su predstavljene neke od metoda koje se koriste za određivanje sadržaja delta ferita u austenitnim čelicima, kao i u metalu zavara.

Professional paper

SUMMARY

Microstructure of an austenitic stainless steels is primarily monophasic, i.e. austenitic. However, the chemical composition of steel can affect on precipitation of a delta ferrite. The presence of the delta ferrite in austenitic stainless steel has a beneficial or detrimental effect on mechanical properties (impact and tensile) as well as on weldability and corrosion resistance. This paper presents some methods of determining the content of delta ferrite in austenitic stainless steels as well as in weld metal.

1. INTRODUCTION

The first discoveries related to the production of stainless steels date back to the 19 th century. The Englishmen Stoddard and Farraday circa 1820 and Frenchman Pierre Berthierin in 1821 noticed that Fe-Cr alloys are more resistant to the influence of certain acids in accordance to other steels. The first studies related to stainless steels were primarily done in a laboratory. Practical application of the stainless steels started in the period from 1910 to 1915 [1]. In this period, the need for material that can be used in different aggressive media and/or at high temperatures but to retain the good mechanical properties led the scientists in different parts of the world to pay attention to solving this problem. Today, 70% of the total production of stainless steels in the world belong to the production of the austenitic stainless steels [2]. These materials are widely used in automotive, petroleum, chemical, construction, food and cryogenics industry as well as other applications due to their excellent properties, such as: a corrosion resistance in different conditions, good mechanical properties, attractive layout of the final product, long service life etc.

Microstructure of the austenitic stainless steels is primarily monophasic, i.e. austenitic, Fig. 1.

However, the precipitation of a delta ferrite is possible depending on the chemical composition, i.e. in dependence of the ratio of alphagene and gammagene elements.

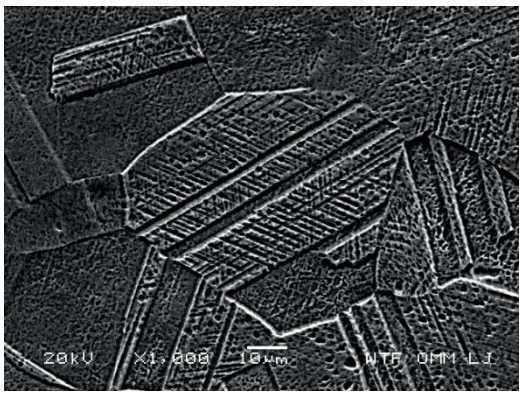

Figure 1 *Microstructure of an austenitic stainless steel Nitronic 60 after a solution heat treatment by SEM (1000x). Etched with a aqua regia [3]*

The solidification of the austenitic stainless steel can start with the crystallisation of the delta ferrite or austenite i.e. there are four modes of solidification, Fig. 2.

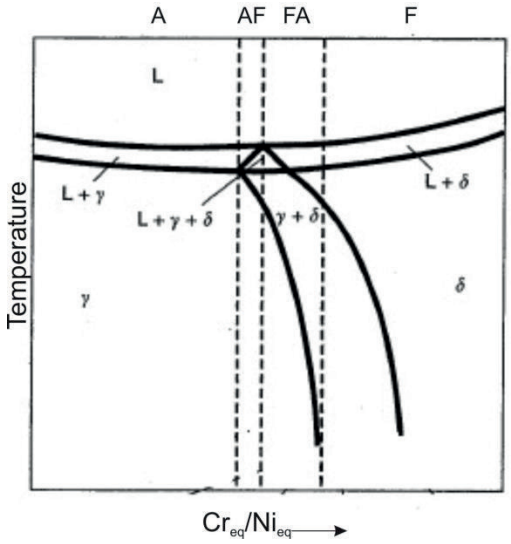

Figure 2 *Vertical section of Fe-Cr-Ni phase diagram, relationship of solidification type [4]*

Modes A (austenite) and AF (austenite-ferrite) where austenite is the primary soldification phase and FA (ferrite-austenite) and F (ferrite) modes where delta ferrite is the primary phase, from Eq. (1) and Eq. (2) follows [3]:

$$L = L + \gamma = L + \gamma + \delta = \gamma + \delta \quad (1)$$
$$L = L + \delta = L + \delta + \gamma = \gamma + \delta \quad (2)$$

As mentioned above, the main alloying elements in austenitic stainless steels can be classified as alphagene and gammagene elements. The alphagene elements (Cr, Si, Ti, Al, Mo, V, Nb, and W) stabilize and promote the formation of delta ferrite, while the gammagene elements (Ni, Mn, C, N and Cu) stabilize and extend the austenite region, and reduce the content of the delta ferrite [5,6].
According to standard ASTM A800/A800M-91, the ferrite is the microstructural constituent with body centered cubic crystal structure and it is ferromagnetic [7]. The delta ferrite occurs during solidification and remains in the microstructure at room temperature, Fig. 3.

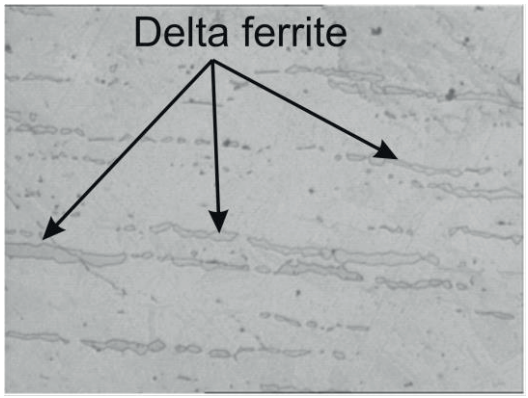

Figure 3 *Optical micrograph of an austenite stainless steel Nitronic 60 with presence of delta ferrite, longitudinal section, 100x Etched with a aqua regia. [3]*

The presence of the delta ferrite slows a grain growth and increases strength properties of the steel. The interphases boundaries are a strong barrier to dislocation motion [8].

The delta ferrite is ductile at room and elevated temperature, but brittle at cryogenic temperatures. However, the delta ferrite has a very important role in welding of the austenitic steel because it decrease sensibility to the appearance of hot cracks. Because many of the weld metals contain delta ferrite, it has important influence on a weld solidification cracking susceptibility. The weld solidification cracking susceptibility is in a function of chemical composition and microstructure. Austenitic steels which solidifed according to AF mode are less susceptibile to cracking than A mode. Reason for this is the presence of a two phase austenite and ferrite mixture along solidification grain boundaries at the end of solidification that resists wetting by liquid films and presents a boundary, which is not straight and smooth, along which crack must

propagate [4]. The delta ferrite content in austenitic welds is usually about 3-8% [9].
However, the content of the delta ferrite decreases through plastic deformation (hotrolling), annealing and the welding method has significant effect on its content too.
During annealing of the austenitic stainless steels at higher temperatures, the intermetallic phases and carbides precipitate from the austenite and/or the delta ferrite. One of the most commonly phases is a sigma phase (σ-phase) [2,10].
The sigma phase is a well known intermetallic compound (FeCr) with a tetragonal crystal structure. In highly alloyed steels, its composition is variable and it is difficult to define this phase in the form of unique formulas [7]. The alloying elements such as chromium, molybdenum, tungsten, vanadium, silicon, manganese, niobium, titanium and tantalum promote the formation of the sigma phase whereas nickel, carbon, nitrogen, cobalt and aluminium hinder its formation [2]. At room temperature this phase is a hard, brittle and nonmagnetic [11], therefore, has a negative effect on mechanical properties especially on toughness and ductility. The presence of the delta ferrite reduces the incubation period of precipitation of the sigma phase. The rate of the sigma phase precipitation from the delta ferrite is about 100 times more rapid than the rate of the sigma phase precipitation directly from austenite [2, 7, 12]. In austenitic stainless steels, precipitation of the sigma phase occurs primarily in the delta ferrite particles but it is possible on grain boundaries too, as well as the so-called "triple points", Fig. 4. [10, 12].

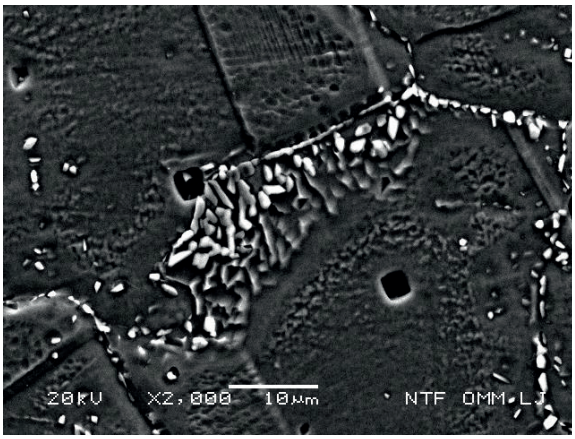

Figure 4 *Microstructure by SEM of a sigma phase in an austenite stainless steel S21800 annealed at 850ºC for 2 hours and water cooling, 2000x. Etched with aqua regia. [3]*

The temperature interval, in which this phase occurs for most austenitic stainless steels, is between 550ºC and 900ºC [2] but decomposes at temperatures above 1000ºC. To get samples free of the sigma phase a heat treatment temperature has to be higher than 1000ºC followed by rapid cooling [11].

Since the content of the delta ferrite is very important in the austenitic stainless steel, because it influence the properties of steel, especially for welding, controll of its content is very often demanded. This paper gives overview of some methods for determination of a delta ferrite content in austenitic stainless steel.

2. METHODS OF DELTA FERRITE DETERMINATION

The significant influence of the delta ferrite on the properties of stainless steels, especially the mechanical and corrosion properties encouraged many researchers to try to find a way to determine the content of the delta ferrite. In his work Bermejo gave a chronological review from the first predictive diagram in 1920 up to the latest mathematical model [13]. Currently used methods for the determination of the delta ferrite in the stainless steels are predictive methods on the basis of chemical composition and measurement methods, i.e. magnetic and metallographic methods.

2.1. Predictive methods

Predictive methods are mainly used to estimate the content of the delta ferrite in castings or weld metal in a preliminary study stage. Determination of the delta ferrite based on chemical analysis is a useful method for controlling the delta ferrite content during melting too. These methods use diagrams as the Schoefer, Schaeffler, DeLong and WRC-1992 diagram where, on the base of a chemical composition, the content of the delta ferrite could be predicted.

Standard ASTM A800/A800M describe procedures and definitions for determination of the delta ferrite content in the castings using by the Schoefer diagram. The Schoefer diagram was first time published in 1973, Fig. 5 [9,13]. This diagram has the coordinates of the composition ratio of the chromium equivalent

to nickel equivalent and ferrite number. In regards to diagram presented in standard ASTM A800/A800M, a multiplier coefficient of molibdenium is 1.

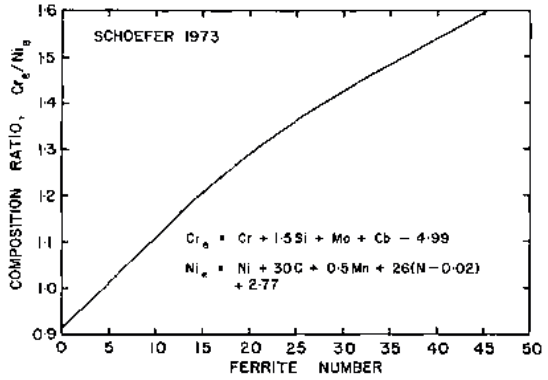

Figure 5 *Schoefer diagram [13]*

Influence of alloying elements on the forming of final microstructure of austenite steel could be expressed by ratio of „chromium equivalent“ (Cr_e) and „nickel equivalent“ (Ni_e) from Eq. 3 as follow [13-15]

$$Cr_{eq}/Ni_{eq}=(\%Cr+1,4\%Mo+1,5\%Si+\%Cb-4,99)/(\%Ni+0,5\%Mn+30\%C+26(\%N-0,02\%)+2,77) \quad (3)$$

Only the presence of the delta ferrite can be determined by using the Schoefer diagram, no other phases such as martensite and austenite. The delta ferrite content determined on the basis of chemical composition depends on the procedures of chemical analysis, i.e. the accuracy of data. Accordingly, the maximum and minimum content of the delta ferrite can be determined on the basis of the standard ASTM A800/A800M [14,15].

The ferite content in weld deposits could be determinated in accordance with the Schaeffler, DeLong or WRC-1992 diagrams. In 1949, Antoine Schaeffler published diagram that represented link between alphagene and gammagene elements and the content of delta ferrite [9,13]. This diagram is known as the Schaeffler diagram, Fig.6.

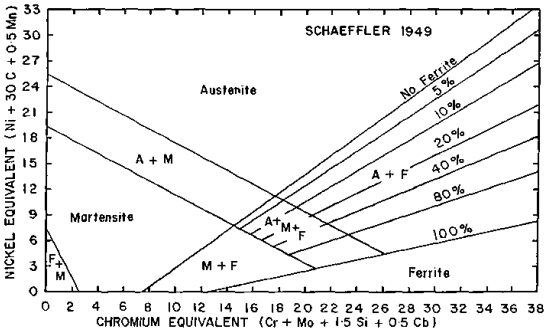

Figure 6 *Schaeffler diagram of 1949 [9]*

The chromium (Cr_{eq}) and nickel (Ni_{eq}) equivalent are also used for the construction of this diagram but in modified form, Eq. 4 and Eq. 5.

$$Cr_{eq}=\%Cr+\%Mo+1,5\%Si+0,5\%Nb \quad (4)$$
$$Ni_{eq}=\%Ni+0,5\%Mn+30\%C \quad (5)$$

Depending on the values of Cr_{eq} and Ni_{eq}, the presence of an austenitic, ferritic, martensitic, phase and a mixture of these phases can be identified. Use of the Schaeffler diagram is limited for the case of a high level of nitrogen in weld [14].

W.T. De Long has studied an effect of nitrogen on the content of the delta ferrite. It is known that nitrogen is a strong austenite phase stabilizer and his influence cannot be ignored. The first DeLong diagrams published in 1956 and included the effect of nitrogen. In contrast to the Schaeffler diagram, an expression of Ni_{eq} is only extended with 30x% N, Eq. 6, [14]

$$Ni_{eq}=\%Ni+0,5\%Mn+30\%C+30\%N \quad (6)$$

The diagram shows the presence of delta ferrite both as percentages (based on metallographic determinations) and as ferrite number "FN" (based on magnetic determination methods). De Long introduced for the first time the term FN in his diagram in 1973, Fig. 7 [13].

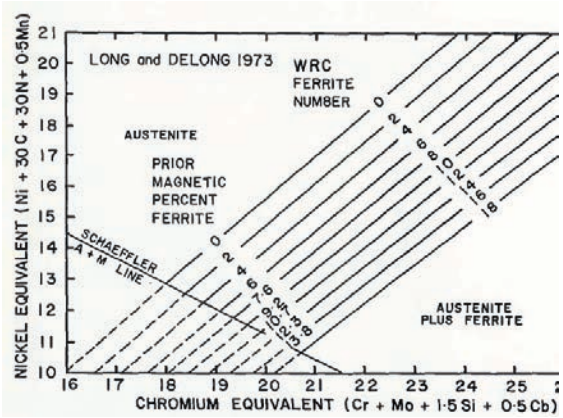

Figure 7 *The Long and DeLong diagram of 1973 [9,13]*

In 1972, the Welding Research Council (WRC) and the International Institute of Welding (IIW) established a procedure for a standardization of the delta ferrite measurements. Ferrite Number (FN) is introduced as a new scale and is defined according to the attractive force between a standard magnet and a set of primary standards made of mild steel substrate electroplated with different thicknesses of nonmagnetic coating. In 1974, the American Welding Society published this procedure as the standard AWS A4.2 and later adopted as ISO 8249 [13].

In 1988, a new diagram called the WRC – 1988 was developed on the base of database of the Welding Research Council for the chemical composition and the FN value. The diagram is obtained using a system of multivariable linear regressions where FN was the dependent variable and every alloying element was an independent variable. Unlike De Long's diagram, which is based only on the AISI-300 austenitic stainless steels, WRC-1988 diagram includes about 923 stainless steels [13]. Eq. 7 and Eq. 8 determine the value of Ni and Cr are equivalent [13, 14]:

$$Cr_{eq}=\%Cr+\%Mo+0{,}7\%Nb \quad (7)$$

$$Ni_{eq}=\%Ni+35\%C+20\%N \quad (8)$$

In 1992, the diagram is modified in a way that takes into account the effect of Cu to Ni_{eq} value, Eq. 9

$$Ni_{eq}=\%Ni+35\%C+20\%N+0{,}25\%Cu \quad (9)$$

The diagram is known as WRC-1992 diagram and used for predicting weld ferrite content and solidification mode, Fig. 8 [14].

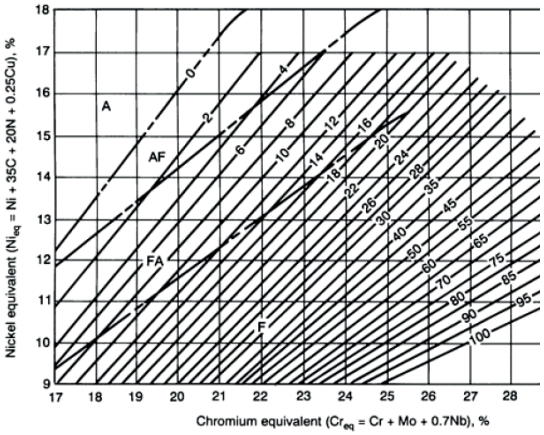

Figure 8 *WRC-1992 diagram [9,13]*

The disadvantage of these method is that it can estimate the interval of the delta ferrite content. Also, in practice for steels for which the diagram predicts delta ferrite content of 0-5%, the actual content is usually lower [16]. In the case of welding, the lack of these methods is that during the welding process may change the chemical composition of the weld or a consumable composition is taken for the calculation instead of the weld metal composition. Also, the lack is that they do not take into account an effect of cooling rate on the content of the delta ferrite and their use is not recommended in the case of high energy welding processes. When the ratio of the heat input to the welding speed increases, the cooling rate decreases, i.e. the delta ferrite content increases [17].

On the basis of many researches, it is concluded that 3 to 8 volume percent delta ferrite is needed to reduce hot cracking susceptibility. However, the existence of the delta ferrite is not enough for preventing of hot cracking. Influencing factors are way of solidification, delta ferrite amount, morphology and distribution. Some investigations show that a primary ferrite solidification mode is necessary. Suutala et al. published a diagram indicating a liquidus projection line as a function of nickel and chromium equivalents, Fig. 9. In the 1980s, Suutala and Kujanpää published a diagram, named the Suutala diagram, predicting cracking susceptibility based on chemical composition, i.e. ratio of the chromium

equivlent and nickel equivlent. Impurity level in steel is very important and tends to increase the cracking susceptibility, particulary content of sulfur and phosphorous [4].

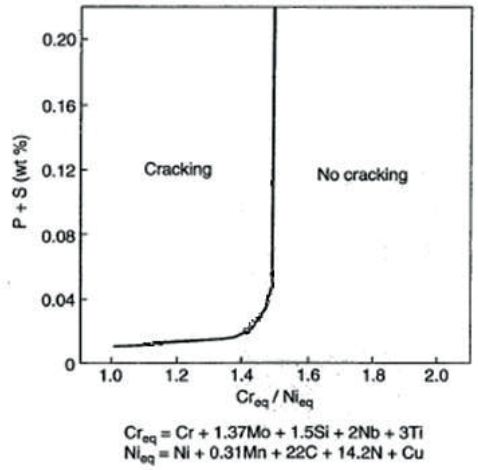

Figure 9 *Suutala diagram for predicting weld solidification cracking [4]*

2.2. Determination using Magnetic Method

Magnetic method as well as a metallographic method is used for determination of the content delta ferrite in heat, product or weld metal of austenite stainless steel and based on attractive force and magnetic permeability. The attractive force method is based on the force required to separate the feromagnetic sample from a stanadardized permanent magnet in the instrument (for example Magne-Gage instrument). The magnetic permeability technique is based on placing a probe coil on the sample that produces a low frequency electric field which interacts with the delta ferrite to generate a magnetic field. The voltage induced by this magnetic field in a separate pickup coil on the probe is proportional to the magnetic permeability and therefore is a direct function of the delta ferrite content [13]. Such as instrument is the Fischer Feritscope, Fig.10. The magnetic test is based on the fact that the austenite is nonmagnetic and the delta ferite is magnetic. This method is nondestructive, quick, and can be used in laboratories as on-site method in production.

Standard ASTM A800 / A800 defines the way of determining the ferrite content by the magnetic method [15]. Before testing, the sample should be prepared, i.e. removed all impurities. If there are impurities, the connection between a probe device and test surface can be interrupted, because the probes for testing are usually small. In this case, the results can be incorrect.

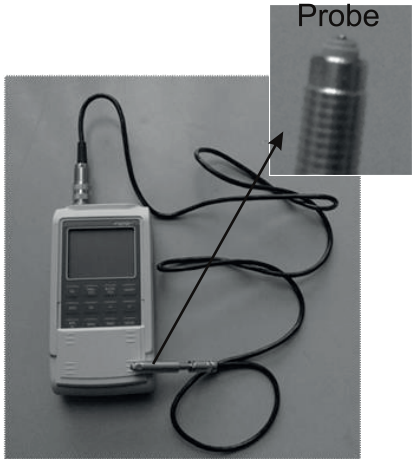

Figure 10 *Feritscope MP30E by a producer Fischer (Helmut Fischer GmbH+Co.KG)*

During the tests, it is necessary to ensure full contact between the probe and the test material. Since the delta ferrite is not homogeneously arranged in the matrix, it is necessary to do more measurements on the same sample. The mean value is determined on the basis of measurements to obtain representative values.

Standard A799/A799M-92 is used for calibration of instruments to be used for estimating the delta ferrite content by magnetic response or measurement of permeability [18].

2.3. Determination using Metallographic Method

A metallographic method has been the main method for the experimental determination of the delta ferrite for long time. Test Method E562-95, i.e. Standard Test Method for Determining Volume Fraction by Systematic Manual Point Count is used to determine the volume fraction of the delta ferrite [19]. According to this method, a test grid or eyepiece reticle with a regluar array of test points is superimposed over the image produced by a light microscope, scanning electron microscope or photograph. The volume percent of the delta ferrite is determined on the base of the total number of grid points and the number of test points falling within the phase (the delta ferrite). The test points present the intersection of the two lines [19]. This test is recommended for castings where the size and morphology of delta ferrite are rougher, but no for weld metals

due to the irregular and thin morphology of the delta ferrite [13].

Determination of the delta ferrite by metallographic analysis depends on the etching techniques used for the identification of the delta ferrite. Usually, agents of etching are aqueous 20% NaOH at 3 V dc for 5 to 20 s, aqueous 10 N KOH at 2.5 V dc about 10 s, Fry's reagent, Marble' reagent, Murakami's reagent, aqua regia, Kalling's etc [20]. Standard metallographic method described in ASTM E3 is used for the preparation of samples [21].

Besides the manual method, standard ASTM E1245 defines the use of automatic image analysis for determining of second-phase [22]. This method using basic stereological procedures to estimate the presence of inclusions or second-phase constituents in metal. Stereology is used to quantify matrix microstructures, as opposed to standard metrology technigues. Microstructural tests are made on a two dimensional polish plane through a three dimensional opaque metal. Stereology converts these 2-D measurements into 3D estimates of microstural paremeters [23].

An example of the metallographic method for determining the delta ferrite content is shown in Fig. 11 for three melts with different delta ferrite content. During the analysis, ten visual fields, at the sample, were photographed and the mean value determined. The software evaluates the share of the ferrite phase on the basis of differences in color. The metallographic examination was performed on Olympus optical microscope with magnification 100x using Olympus software for phase analysis.

Fig. 12 presents results of determination of the delta ferrite in austenitic stainless steel Nitronic 60. Five melts with different chemical composition were tested. The chemical composition of the samples was in accordance with ASTM A276-96 standard. The samples were tested after the solution heat treatment (heating at 1020 ^{0}C and cooling in water). Three methods (the predictive, magnetic and metallographic methods) were used for the determination of the delta ferrite content.

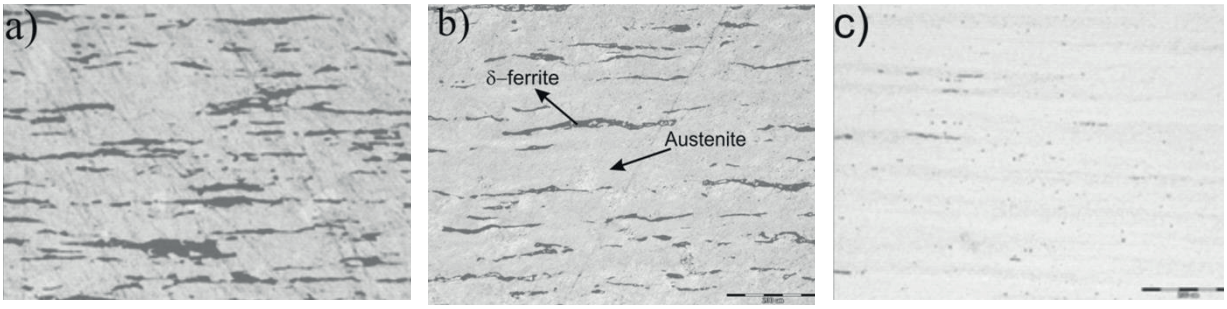

Figure 11 *Analysis of the delta ferrite (DF) content using an optical microscope with Olympus software for the phase analysis for the three melts of steel Nitronic 60: a) 10,56% DF, b) 5,2% DF and c) 0,31% DF [3]*

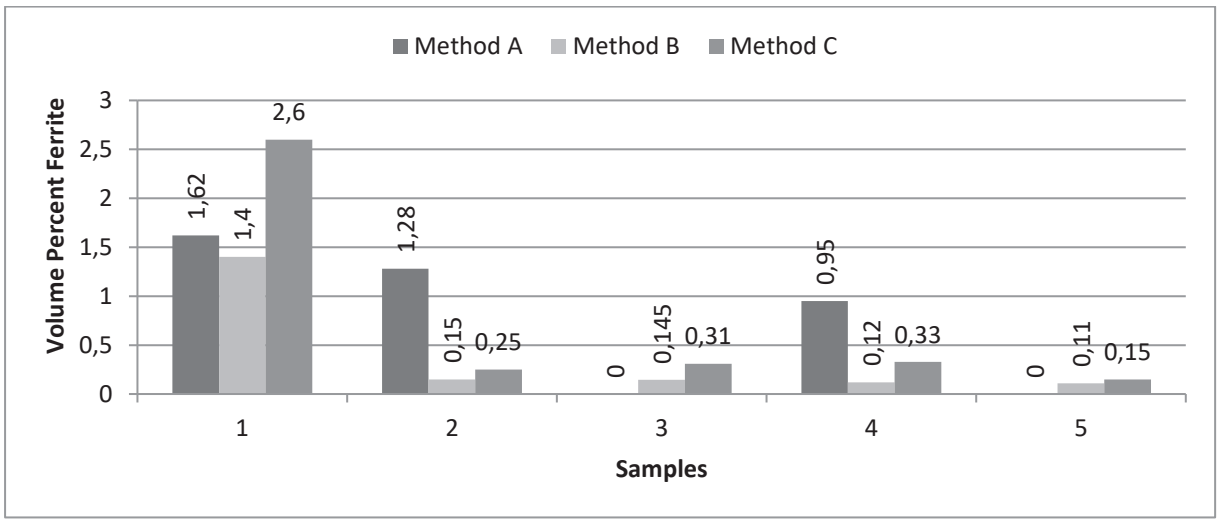

Figure 12 *Volume Percent Ferrite determined by Method A - the Schoefer diagram, Method B - Magnetic method and Method C - Metallographic method*

The predictive tests used the Schoefer diagram (Method A). A volume percent ferrite was determined on the basis of ratio (Cr_e / Ni_e) and using diagrams and tables (standard ASTM A800/A800M). The method defines a minimum, medium and maximum value of the volume percent ferrite due to the possibility of errors in the chemical analysis. Since the content of the delta ferrite is a quite small and a minimum content is 0.00 vol % the maximum volume percent ferrite is taken as reference.

The magnetic method (Method B) is other method used for testing. The test was done in accordance with the standard ASTM A800 / A800M by Fischer Feritscope.This instrument is used for non-destructive measurement of the delta ferrite content in a range of 0.1 to 110 FN or 0.1 to 80% Fe in austenitic and duplex steel. The Feritscope measures according to the magnetic induction method. A magnetic field generated by coil enters into interaction with the magnetic components of the sample. The changes in the magnetic field induce a voltage proportional to the ferrite content in a second coil. This voltage is then evaluated [24, 25].

For the metallographic methods, the samples were cutting, grinding, polishing and etching in the Kalling's solution. Analysis of microstructure was done by Olympus optical microscope with magnification 100x equipped with Olympus software for phase analysis [24].

From the Fig. 12, it could be seen that there is not signifficant difference between results for the three different methods of testing.

3. CONCLUSIONS

Austenitic stainless steels are primarily monophasic, i.e. austenitic. However, the precipitation of a delta ferrite is possible depending on the chemical composition. The delta ferrite occurs during solidification and remains in the microstructure at room temperature. It is ductile at room and elevated temperature, but brittle at cryogenic temperatures and protects from the appearance of hot cracks in welds. During the annealing of the austenitic steels, there is a possibility of transformating of the delta ferite in a sigma phase. The sigma phase remains in the steel if a temperature of annealing is not more than 1000^0C and if a cooling is not very fast. This phase is hard, brittle and has a negative effect on mechanical properties, especially on toughness and ductility.

A control of delta ferrite content in austenitic steels is of a great importance, because of its influence on the mechanical and corrosion properties of steel, weldability and thermal stability. The content of delta ferrite can be determined using predictive, metallographic or magnetic method.

Predictive method is very simple and fast method, but it uses a chemical composition as a base for predicting the content of delta ferrite. The use of chemical composition alone could not be sufficient, because the small change in composition, or a wrong composition, could influence results.

Metallografic method is quite precise, but has some disadvantage. Method is destructive, i.e. requires taking a sample from weld, needs

more time for preparing a sample for metallographic examination (grinding, polishing, etching), reproducibility is poor etc. Magnetic method shows the best results and thanks to its advantages is widely adopted and standardized. This method is quite precise, non destructive, fast and repeatable.

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