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THE EFFECT OF ANNEALING ON MICROSTRUCTURE OF STEEL NITRONIC 60

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ABSTRACT

Nitronic 60 (UNS S21800) is a highly alloyed austenitic stainless steel with the increased content of manganese and silicon which has good mechanical and corrosion properties at high temperatures and loads. This paper presents the results of investigations of the influence of annealing process on the microstructure of steel Nitronic 60. Heat treatment consisted of annealing from 400 to 850^{0}C for four hours followed by cooling in the water. The light and scanning electron microscope (with EDS analysis) were used for microstructure analysis. Analysis showed presence of sigma phase at higher temperatures. The sigma phase was precipited mainly at the austenite grain boundaries, δ/γ interface boundary and δ ferrite phase. Hardness testing showed increasing of hardness with precipitation of sigma phase.

Key words: austenite stainless steel, austenite, δ phase, σ phase

1. INTRODUCTION

Microstructure stability is the most important requirements to obtain proper mechanical properties of an austenitic stainless steel (ASS) [1]. To achive a stable microstructure, the samples are usually solution heat treated at temperature range betveen 1000 to 1120^{0}C and water quenched [2]. Microstructure of Nitronic 60 is primarily monophasic ie. austenitic. However, precipitation of the delta ferrite (δ-ferrite) in the austenite matrix is possible depending of the chemical composition [3, 4]. During annealing at 500-900^{0}C the intermetallic phases and carbides precipitate from the austenite and/or the δ-ferrite [2,5]. One of the most commonly phases in ASS is a sigma phase (σ-phase) [2,6]. Depending on the heat treatment temperature, the δ-ferrite can be transformed in the the σ-phase [5,6,7,8,9,10]. The σ-phase is an intermetallic compound with a complex tetragonal crystal structure. The chemical composition of this phase varies considerably and is therefore difficult to define this phase in the form of unique formulas. At room temperature this phase is a hard, brittle and nonmagnetic [10] therefore has a negative effect on mechanical properties especially on toughness and ductility and corrosion resistance [11]. Intermetallics such as σ-phase are usually sources of failure in high-temperature materials. The temperature interval, in which this phase occurs for most commercial steels, is between 590^{0}C and 870^{0}C but decomposes at temperatures above 1000^{0}C. To get samples free of the σ-phase, the heat treatment temperature has to be higher than 1000^{0}C followed by rapid cooling. Usually, the σ-phase precipitates after long term aging at high temperatures (for example 10 000-15000 hours at 600^{0}C) but different analysis shows that it can be form even for shorter period of annealing [12]. The aim of the research presented in this paper is investigation of annealing effect from 400 to 850^{0}C on steel microstructure.

2. EXPERIMENTAL WORK

Chemical composition of the tested austenite stainless steel Nitronic 60 is given in Table 1. The heat treatment was done using samples in the solution annealed condition. The steel was solution annealed at 1020^{0}C for 1 hour followed by water quenching. The samples were heat treated at 400, 550, 650, 750 and 850^{0}C for 4 hour and cooled in water. The microstructural analysis was carried out by a light microscope and scanning electron microscope (SEM) equipped with energy dispersive x-ray spectrometer (EDS). The samples for microstructure analyzing were prepared with standard grinding and polishing techniques and etched by an aqua regia. Hardness was measured by Vickers test (HV_{10}).

Table 1.Chemical composition of steel Nitronic 60.

Melt	Chemical composition , wt/%							
	C	Si	Mn	Cr	Ni	P	S	N
V1702	0.04	3.8	7.4	17.3	8.6	0,007	0.011	0.158

3. RESULTS AND DISCUSION

The microstructure analysis of sample annealed at 400 ^{0}C by the light miroscope showed a typicaly an austenite microstructure with the polygonal grains and twins. The presence of the delta ferrite was noticed, too. The δ-ferrite islands are elongated in the rolling direction. Precipitation of the δ-ferrite is mainly at grain boundaries**.** EDS analysis also confirmed the presence of nonmetallic inclusions MnS i Al_2O_3. The sample annealed at 550 ^{0}C retained polygonal in shape of grains with twins but the beginning of recrystallization was observed in some places in the sample, Figure 1a. The new formed grains are very small and more rounded in shape compared to the initial austenitic microstructure. Figure 1b. shows the presence of the globular shape precipitates at austenite grain boundaries of the recrystallizated microstructure.

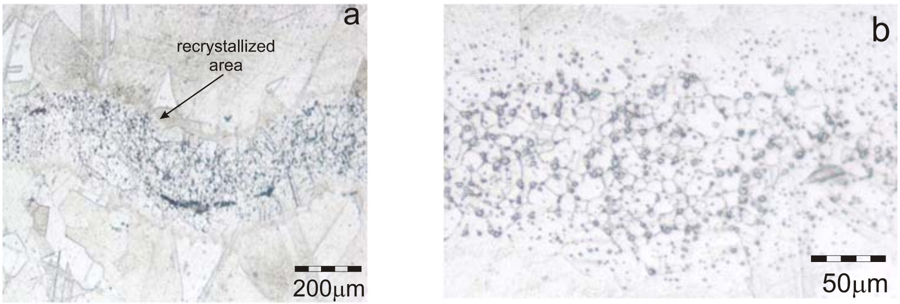


Figure 1. Microstructure of the sample annealed at 550 ^{0}C.

Also, the precipitation presence was noticed in an area near to the δ-ferrite. EDS point analysis showed that the precipitates are enriched in Cr, Si, and Fe, Figure 2.

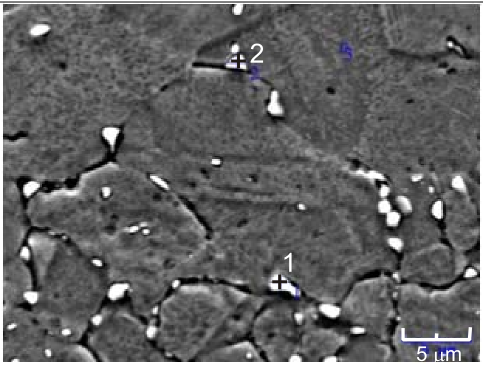
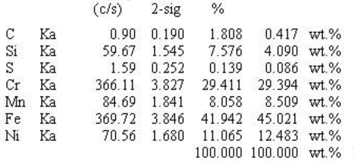


Figure 2. EDS point analysis of the sample annealed on 550^{0}C for 4 hours.

Spectar 1



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Elt.  Line  Intensity  Error   Atomic     Conc
            (c/s)      2-sig   %

C     Ka       0.90    0.190    1.808    0.417  wt.%
Si    Ka      59.67    1.545    7.576    4.090  wt.%
S     Ka       1.59    0.252    0.139    0.086  wt.%
Cr    Ka     366.11    3.827   29.411   29.394  wt.%
Mn    Ka      84.69    1.841    8.058    8.509  wt.%
Fe    Ka     369.72    3.846   41.942   45.021  wt.%
Ni    Ka      70.56    1.680   11.065   12.483  wt.%
                              100.000  100.000  wt.%
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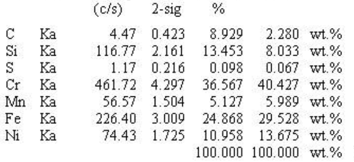
Spectar 2



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Elt.  Line  Intensity  Error   Atomic     Conc
            (c/s)      2-sig   %

C     Ka       4.47    0.423    8.929    2.280  wt.%
Si    Ka     116.77    2.161   13.453    8.033  wt.%
S     Ka       1.17    0.216    0.098    0.067  wt.%
Cr    Ka     461.72    4.297   36.567   40.427  wt.%
Mn    Ka      56.57    1.504    5.127    5.989  wt.%
Fe    Ka     226.40    3.009   24.868   29.528  wt.%
Ni    Ka      74.43    1.725   10.958   13.675  wt.%
                              100.000  100.000  wt.%
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It confirmed assumtion that precipitated phase is σ-phase. In fully austenitic alloys, like Nitronic 60, the σ phase forms from the austenite along grain boundaries [11]. The presence of the δ-ferrite reduces the incubation period of precipitation of the σ-phase. The rate of the σ-phase precipitation from the δ-ferrite is about 100 times higher than the rate of the σ-phase precipitation directly from austenite [3]. The amount of the σ phase was increased with increasing of annealing temperature at 650 ^{0}C. The σ phase was present at the austenite grain boundaries, inside of austenite grain and δ/γ interphase boundary, Figure 3. due to higher boundary energy and defects that are concentrated there. Therefore, the precipitation of σ phase takes place preferentially at δ/γ boundary, growing toward interior of delta-ferrite grain [11]. The σ phase has a globular in shape. The σ phase usually precipitates in two types of morphologies globular and dendritic. Literature sources shows that the globular shape is stable, while the dendritic shape is unstable one. Also, the dendritic structure is brittle, while, the globular structure is ductile [11,13]. The δ-ferrite transformation in the σ phase was specifically pronounced at the annealing temperature 750^{0}C. The σ phase has a cellular shape, Figure 4. The cellular shape precipitation presented the eutectoid decomposition from δ-ferrite into σ and secondary austenite phases [11]. Also, the σ phase in globular form was precipitated at the austenite grain boundaries and within the grains, Figure 4. The σ phase was present in the microstructure of the sample annealed at 850 ^{0}C. In all samples the grain size is quite uneven. EDS analysis of the all samples annealed from 550 to 850 ^{0}C show that the Cr and Si content in the σ-phase were higher than that of the austenite-phase. Generally, δ-ferrite and σ-phase are Cr-rich. The σ-phase preferred to precipitate at a higher Cr content region. Si has an important role in transformation δ ferrite and acts as strong stabilizers for δ-ferrite [11]. Figure 5. shows EDS maps of the σ phase and confirm that the σ phase is enriched in Cr, Si and Fe. Also, the EDS analysis of the σ phase (Figures 2.) showed a slightly higher concentration of Ni (ca. 13%) compared to the austenitic where the content was of 9-10 wt. % what is not clearly seen in the case of EDS map analysis.

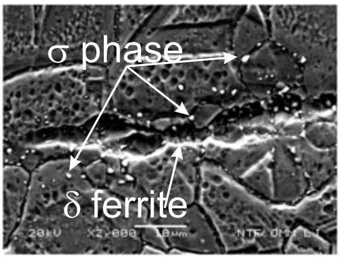


Figure 3. SEM analysis of the sample annealed on 650^{0}C for 4 hours and water quenched

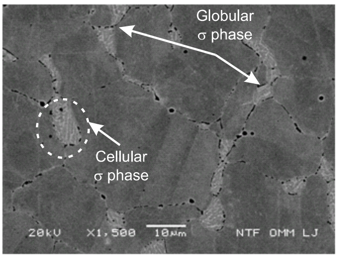
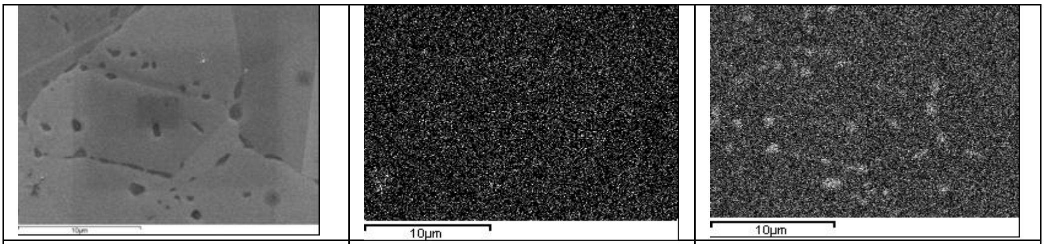
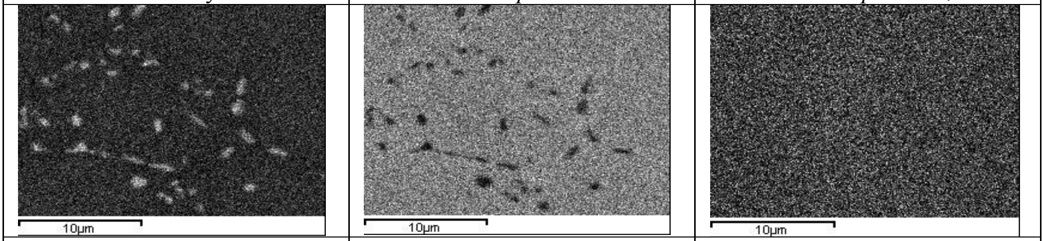


Figure 4. SEM analysis of the sample annealed on 750^{0}C for 4 hours and water quenched



SEM analysis | *EDS map C Ka1* | *EDS map Si Ka1,*



EDS map Cr Ka1, | *EDS maa Fe Ka1,* | *EDS mapaNi Ka1*

Figure 5. EDS maps of the sample annealed on 850^{0}C for 4 hours and water quenched

4. TESTING OF HARDNESS

The results of the hardness testing are shown in Table 2. The testing was done in accordance with the standards EN ISO 6507-1/2007 and EN ISO 6508-1/2007 on the sample in the solution annealed condition and the samples annealed at 400, 550, 650, 750 and 850 ^{0}C. The sample annealed at 400^0C had the lowest value of hardness as like as the sample in the solution annealed condition (initial state). The change of microstructure was not noticed at the sample annealed at 400^0C. From Table 2., it can be seen increasing of hardness for the sample annealed at 550^0C because of the precipitation of the σ phase which is hard and brittle. The samples annealed at higher temperatures from 650^0C to 850^0C show no significant change in hardness.

Table 2. The results of the hardness testing

Heat treatment	**Hardness [HV 10]**
1020^0C/1 hour/water (initial state)	207
400^0C/4 hour/water	208
550^0C/4 hour/water	232
650^0C/4 hour/water	228
750^0C/4 hour/water	223
850^0C/4 hour/water	236,8

5. CONCLUSIONS

After the solution annealing at temperature of 1020 ^{0}C for 1 hour followed water quenching was obtained austenite microstructure with precipitated δ ferrite. Annealing at 400 ^{0}C did not cause the change of the microstructure what was confirmed by the hardness testing. The hardness was the same as in initial state. The recrystalisation process was started at 550 ^{0}C and precipitation of the σ phase on the grain boundaries of recrystallized microstructure. At temperatures above 550 ^{0}, the precipitation of the σ phase was not present only on the austenite grain boundaries also within the grains and at the δ / γ interphase boundary. At temperature of 750^0C starts eutectoid decomposition from δ-ferrite into σ and secondary austenite phases. In general, for this alloy, the σ phase forms over the range of 550 to 850 ° C. The σ phase precipitates on grain boundaries, inside grain and the δ / γ boundary interphase are globular in shape while the σ phase formed from the eutectoid decomposition of the δ-ferrite is cellular in shape. The globular shape is more acceptable because it is a stable. The precipitation of the σ phase at higher temperatures was not have significant influence on the hardness. The difference in hardness between the different treatment temperatures is not so high.

6. REFERENCES

[1] J.Janovec, B.Šuštaršić, J.Medved, M.Jenko, Materiali in tehnologije 37 (2003) 6

[2] R.L.Plaut, C.Herrera, D.M.Escriba, P.R.Rios, A.F.Padilha, Materials Research vol.10 No.4 (2007)

[3] A. Gigović-Gekić, Mirsada Oruč, Mirko Gojić:Determination of the content of delta ferrite in austenitic stainless steel Nitronic 60, 15th International Research/Expert Conference TMT 2011, Prague, Czech Republic, 12-18 September 2011

[4] M. Oruč, M. Rimac, O. Beganović, A. Delić: New materials as base for development of modern industrial technologies, 13th International Research/Expert Conference TMT 2009 Hammamet, Tunisia, 16-21 Octo. 2009

[5] A.F.Padilha, P.R.Rios, ISIJ International 42 (2002)No.4, pp. 325-337

[6] H.S. Khatak, B. Raj: Corrosion of Austenitic Stainless Steels Mechanism, Mitigation and Monitoring, 2002.

[7] F.Tehovnik, F.Vodopivec, L.Kosec, M.Godec, Materiali in tehnologije 40 (2006) 4

[8] J.C.Tverberg, The Role of Alloying Elements on the Fabricabiliy of Austenitic Stainless Steel, [cited:01.04.2009.] Avaible from World wide Web: www.csidesigns.com/tech/fabtech

[9] F.Tehovnik, B.Arzenšek, B.Arh, D.Skobir, B.Pirnar, B.Žužek, Materiali in tehnologije 45 (2011) 4,

[10] S.Kožuh, M.Gojić, L.Kosec, RMZ-Materials and Geoenvironment 54 No.3 (2007)

[11] Mohammad Hosein Bina (2012). Homogenization Heat Treatment to Reduce the Failure of Heat Resistant Steel Castings, Metallurgy - Advances in Materials and Processes, Dr. Yogiraj Pardhi (Ed.), ISBN: 978-953-51-0736-1, InTech, DOI: 10.5772/50312. http://www.intechopen.com/books/metallurgy-advances-in-materials-and-processes/homogenization-heat-treatment-to-reduce-the-failure-of-heat-resistant-steel-castings

[12] T.Sourmil, Materials Science and Technology, vol.17 (2001), p.1-14

[13] Chih-Chun Hsieh andWeiteWu: Overview of Intermetallic Sigma (σ) Phase Precipitation in Stainless Steels, ISRN Metallurgy Volume 2012, www. downloads.hindawi.com/isrn/metallurgy/2012/732471.pdf