IMPACT OF SOLUTION ANNEALING ON PROPERTIES OF DUPLEX STEEL

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Abstract

This paper deals with the impact of solution annealing on the properties of X2CrNiMoN2253 duplex steel. Experimental specimens were 100×100×150 mm forged and air-cooled pieces, which were solution-annealed at several temperatures changed in 20 °C steps between 1020 and 1120 °C.

Mechanical tests conducted on these processed specimens included tension tests and Charpy impact tests at room temperature and at -46 °C. The results were compared with the requirements stipulated in the EN 10088-3 standard and with the minimum requirements for X2CrNiMoN2253 forgings for coastal oil and gas extraction set out in technical delivery conditions.

Microstructures of the specimens were explored using optical and electron microscopes and EBSD analysis. EBSD measurement showed significant ferrite texture in some specimens, whereas no signs of texture were detected in austenite in any of the samples. The causes of the ferrite texture were investigated and its impact on properties of the duplex steel was explored.

Keywords: Duplex steel, solution annealing, EBSD analysis

1. INTRODUCTION

The history of duplex or two-phase austenitic-ferritic stainless steels is almost as long as that of stainless steels. However, the interest of industry in the first group of steels has been increasing recently [1-4]. This is the case mainly in those applications where austenitic steels do not guarantee trouble-free and safe operation, particularly in environments where stress-corrosion cracking may occur. The ferrite/austenite ratio ranges from 30 to 50 % and is mainly governed by the content of ferrite formers but also depends on heat treatment history [5]. Austenitic-ferritic steels normally contain 0.02 % C and 0.25 % N, and various levels of Cr, Ni and Mo but there are a number of other variants with different levels of alloying additions [6]. Duplex stainless steels find use in numerous applications in chemical, petrochemical, paper and power industries thanks to their attractive combination of good mechanical properties and high corrosion resistance [7, 8]. Their resistance to uniform corrosion is similar to that of austenitic steels but their strength is much higher, in part due to the addition of about 0.15 % nitrogen.

The temperature schedule of the production of duplex steel must reflect the risk of formation of undesirable intermetallic phases in the region below 950 °C [9]. If the temperature of a forged part decreases below this value, a microstructural change occurs which is accompanied by formation of intermetallic phases [10, 11]. Intermetallics considerably reduce mechanical properties and corrosion resistance of final products of duplex steels [12-14]. Since the aforementioned temperature condition cannot be met in all production routes for forged duplex steel parts, solution annealing is employed as either an in-process or post-forming step [15]. This paper deals with the impact of various solution annealing temperatures on the microstructure and mechanical properties of duplex steel.

2. EXPERIMENTAL MATERIAL

The experimental specimens were forged pieces of 100 × 100 × 150 mm size of X2CrNiMoN2253 steel made by the company ŽĎAS, a.s. Their chemical composition was in accordance with the EN 10088-3 standard. All the specimens were forged using the same sequence, and subsequently cooled in air. At COMTES FHT a.s., the forged pieces were solution-annealed at various temperatures between 1020 °C and 1120 °C in steps of 20 °C. After annealing, they were quenched in water. The specimens were placed into a furnace at 500 °C, soaked for one hour, and then heated at a rate of 100 °C / hour to the annealing temperature. They were held for 4 hours and then quenched in water. Mechanical testing and metallographic analysis were then carried out on specimens made from these treated forged pieces.

3. MECHANICAL TESTING

The minimum requirements for mechanical properties of the X2CrNiMoN2253 steel are set out in the EN 10088-3 standard. Besides those, further specifications given in customer technical delivery conditions (TDC) frequently apply to the manufacture of forgings for coastal production oil and natural gas. **Table 1** gives a concrete example of typical specifications for forged parts of X2CrNiMoN2253 duplex steel for coastal production of oil and natural gas [13].

Upon agreement with the feedstock manufacturer, tension tests, notch toughness tests at ambient temperature and notch toughness tests at the reduced temperature of -46 °C were carried out only in the longitudinal direction.

Table 1 Mechanical property specifications for X2CrNiMoN2253 steel according to typical TDC [13]

Test type	**Tension test**				**Notch toughness test**			
Testing temperature	+20 °C				-46 °C			
Minimum values according to TDC	Re	Rm	A	Z	Longitudinal		Transverse	
					Individual	Mean value	Individual	Mean value
	[MPa]	[MPa]	[%]	[%]	[J]	[J]	[J]	[J]
	450	620	25	45	35	45	20	27

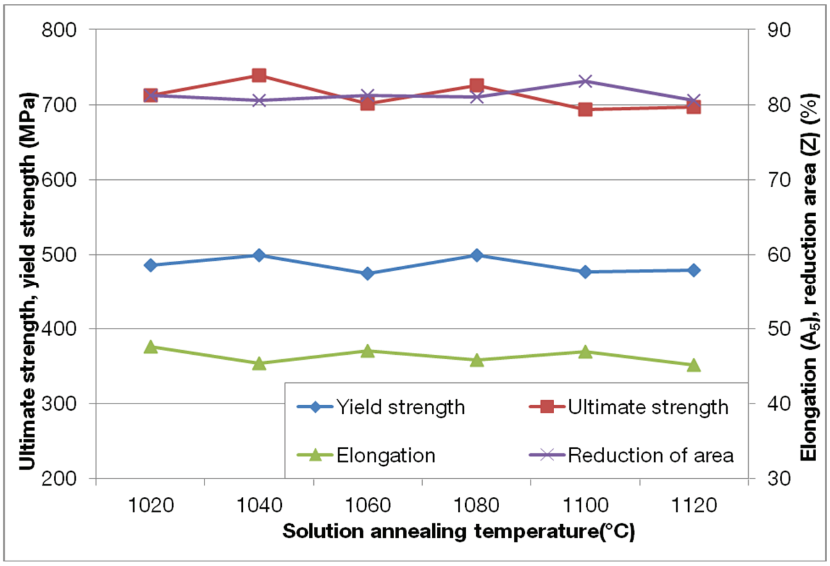

Figure 1 Results of the ambient temperature tension test

The tension test data plotted in **Figure 1** are mean values calculated from results for three individual test pieces. **Figure 1** shows that mechanical properties (ultimate strength R_m, yield strength R_e, elongation in 5D A_5, and reduction of area Z) do not vary considerably with the temperature of solution annealing. The tension test data indicate no distinct trend of mechanical properties with respect to the solution annealing temperature. The values of yield strength, ultimate strength, elongation and reduction of area in **Figure 1** are sufficiently higher than the minimum values specified in the TDC (**Table 1**) and, by the same token, the EN10088-3 requirements.

As with the tension test data, the notch toughness values at ambient temperature show no distinct trend indicating a dependence on the solution annealing temperature (**Figure 2**). The best average values were obtained for the temperatures of 1040 °C and 1080 °C. In the latter case, the notch toughness exceeded 300 J in two specimens which means that the pendulum did not rise after the impact. The specimens treated at a single annealing temperature showed a relatively large scatter of notch toughness values. For instance, the values for those annealed at 1100 °C differed by almost 100 J. Nevertheless, all values were considerably higher than the required minimum of 100 J prescribed by EN 10088-3.

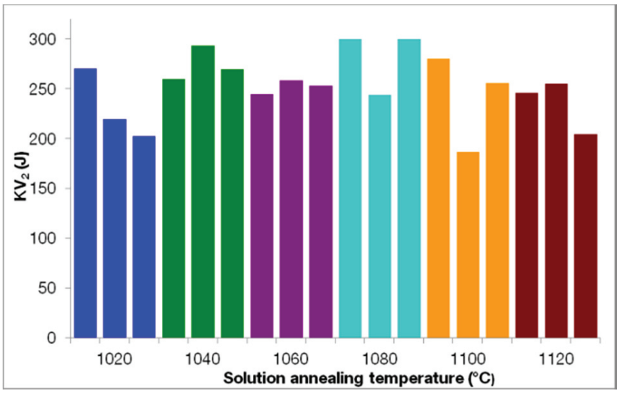

Figure 2 Results of the ambient temperature notch toughness test

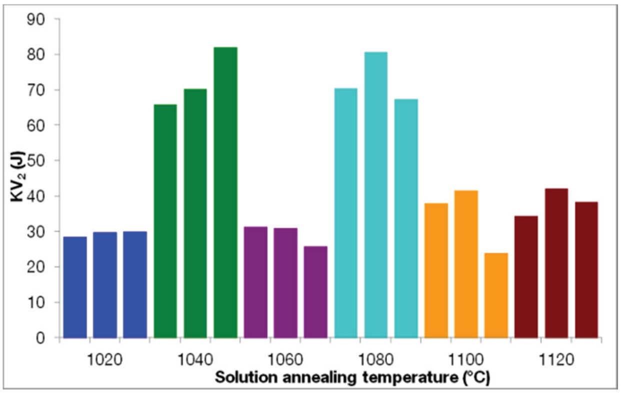

Figure 3 Results of the notch toughness test at the reduced temperature -46 °C

The notch toughness values measured at -46 °C vary greatly with the solution annealing temperature. For the temperatures of 1020, 1060, 1100 and 1120 °C, the results are very low, less than half of those for 1040 and 1080 °C (**Figure 3**). The trend found at the low testing temperature is of the same nature but much more pronounced than that at ambient temperature. The specimens of the material annealed at 1020, 1060, 1100 and 1120 °C failed to meet the above-described TDC. Only the specimens annealed at 1040 and 1080 °C did.

4. METALLOGRAPHIC ANALYSIS

One metallographic specimen was prepared in longitudinal direction from each specimen solution-annealed at a particular temperature. Phase fractions and grain size were measured on the specimens. No intermetallic particles were found in the microstructure under optical microscope or in scanning electron microscope (see **Figures 4, 5**).

As this material is duplex steel that consists of two phases, ferrite and austenite, a separate grain size value was measured for each phase. To this end, the microstructure was revealed by electrolytic etching with 60 % nitric acid (**Figure 5**) and the intercept method was employed. Phase fractions were determined by quantitative image analysis upon etching with Beraha II with an addition of $K_2S_2O_5$ which highlights individual phases.

The grain sizes of both austenite and ferrite were found to increase with the solution annealing temperature (**Figure 4**). This increase is relatively small and has no significant impact on mechanical properties.

The evaluation of phase fractions revealed that the proportion of austenite decreased with increasing temperature of solution annealing. At 1020 °C, the austenite fraction was almost 54 %. At 1120 °C, it was no more than 47 %. The fraction of ferrite increased inversely. However, the differences between the fractions of both phases are not significant.

Contributing to these microstructural changes was the dissolution of small austenite grains embedded in large ferritic regions. Upon solution annealing at 1020 °C, these small grains were frequent in the microstructure. However, upon 1120 °C, practically all of them ceased to exist.

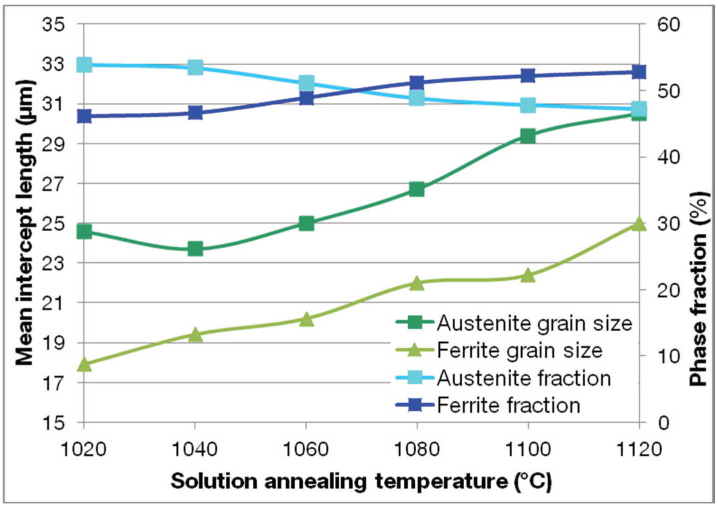

Figure 4 Grain size and phase fraction vs. annealing temperature

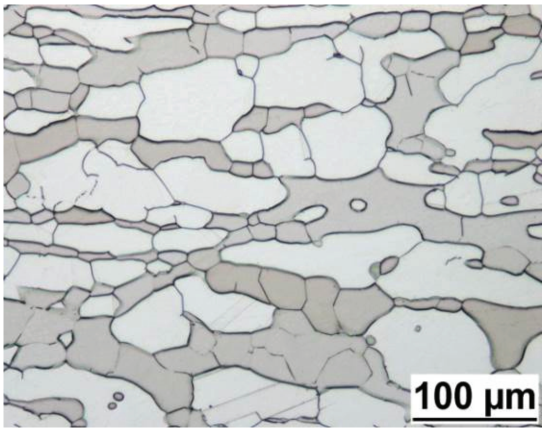

Figure 5 Micrograph of the specimen annealed at 1100 °C upon etching with 60 % nitric acid

4.1. EBSD analysis

Specimens for EBSD analysis were taken from a location just beneath the fracture surface in the test pieces for ambient temperature notch toughness testing (approx. 1.5 mm from the fracture). Textures were examined by means of EBSD in four test pieces. For the annealing temperature of 1040 °C, it was the piece no. 2 which showed the notch toughness of 293 J. For the annealing temperature of 1080 °C, it was the piece no. 1 which showed the notch toughness of 300 J. For the annealing temperature of 1100 °C, two test pieces were studied: no. 1 which showed 280 J, and no. 2 which exhibited a rather low value of 186 J.

In the specimen 1100-2, which had a notch toughness of 186 J, the fracture was of mixed type, comprising both brittle and ductile regions. The other specimens suffered fully ductile fractures. Cleavage facets in the 1100-2 specimen exhibited tongue patterns and were surrounded by ductile fracture surface with dimples. Cleavage occurred in ferritic regions, whereas the dimples are probably associated with austenite areas.

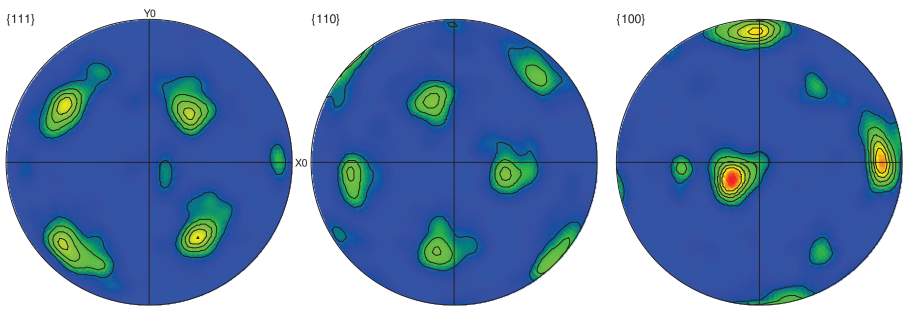

Figure 6 The specimen upon annealing at 1100 °C which had a notch toughness of 28 J. Pole figure for ferrite. Y0 denotes the longitudinal axis of the forged piece and of the notch toughness test piece

The EBSD analysis was carried out in a JEOL JSM-7400 microscope equipped with an EBSD camera from OXFORD Instruments. EBSD mapping at the acceleration voltage of 25 kV and texture analysis were carried out on all specimens. Whereas the specimens annealed at 1040 and 1080 °C (which met the TDC) exhibited no texture, those annealed at 1100 °C (which failed to meet the TDC) showed notable texture in ferrite (**Figure 6**) and no texture in austenite (**Figure 7**).

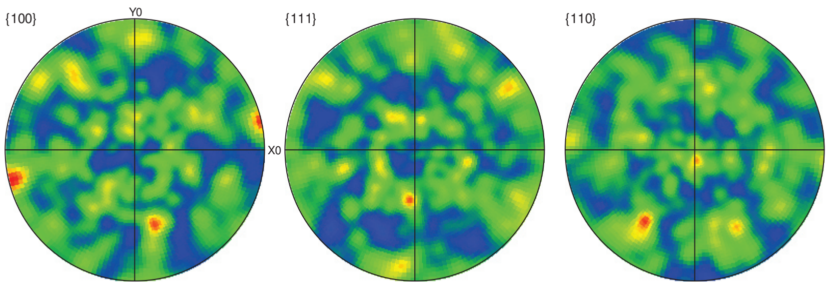

Figure 7 The specimen upon annealing at 1100 °C which had a notch toughness of 280 J. Pole figure for austenite. Y0 denotes the longitudinal axis of the forged piece and of the notch toughness test piece.

5. CONCLUSION

Mechanical testing and metallographic analysis were carried out on 6 forged pieces made of the X2CrNiMoN2253 material which were solution-annealed at various temperatures between 1020 and 1120 °C. The mechanical properties determined by tension testing (ultimate strength R_m, yield strength R_e, elongation in 5D A_5, and reduction of area Z) do not vary considerably with the temperature of solution annealing. As with the tension test, it is impossible to detect any distinct trend of the varying impact energy levels from the notch toughness test in dependence on the solution annealing temperature.

The comparison between the measured mechanical properties and minimum values set out in the EN10088-3 standard, which defines the **m**inimum requirements for mechanical properties of the X2CrNiMoN2253 **steel,** suggests that all mechanical properties measured are sufficiently higher than the required values**.** The notch toughness values measured at the reduced temperature of -46 °C vary greatly with the solution annealing temperature. For the temperatures of 1020, 1060, 1100 and 1120 °C, the results are very low, less than half of those for 1040 and 1080 °C. The specimens of the material annealed at 1020, 1060, 1100 and 1120 °C failed to meet the TDC. The resulting microstructures in all specimens which were analysed are practically identical. They consist of ferrite and austenite with no apparent intermetallic phases. Grain size and phase fractions were determined in the annealed specimens. It was found that the grain sizes of both austenite and ferrite increased with the solution annealing temperature. However, this increase was very small. In addition, the fraction of austenite decreased with increasing solution annealing temperature, whereas the fraction of ferrite increased accordingly. The differences between the fractions of both phases were small as well. All differences between microstructures obtained at various solution annealing temperatures were minute. They probably had no effect on the mechanical properties of the forged pieces.

EBSD examination of specimens annealed at 1100 °C revealed the presence of texture in ferrite grains, whereas the austenite ones were texture-free. This was found in both the 1100-2 specimen, which had a low ambient-temperature notch toughness value, and the 1100-1 specimen with a much higher ambient-temperature notch toughness level. The notch toughness values of these specimens measured at -46 °C were approximately half those of specimens annealed at 1080 °C and 1040 °C. The specimens of the material annealed at 1080 °C and 1040 °C exhibited no texture in ferrite or austenite. One can therefore conclude that the underlying aspect of the drop in notch toughness in the specimens annealed at 1100 °C and, by extension, those annealed at 1020, 1060 and 1120 °C is the presence of texture in ferrite. Hence, the results of mechanical testing were not markedly affected by the solution annealing temperature but the formation of texture during forging of the pieces.

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REFERENCES

[1] LILJAS, M. 80 years with duplex steels: a history review and prospects for the future. In *6th European Stainless Steel Conference, Science and Market.* Helsinki, Finland, 2008, pp. 535-540.

[2] MAKI, T., FURUHARA, T., TSUZAKI, K. Microstructure development by thermomechanical processing in duplex stainless steel. *ISIJ International*, 2001, vol. 41, pp. 571-579.

[3] NILSSON, J.O., Super duplex stainless steels. *Material Science and Technology*, 1992, vol. 8, pp. 685-700.

[4] DUPOIRON, F., AUDOUARD, JP. Duplex stainless steels: a high, mechanical properties stainless steels family. *Scandinavian Journal of Metallurgy*, 1996, vol. 25, pp. 95-102.

[5] REICK, W., POHL, M., PADILHA, A.F. Recrystallization-transformation combined reactions during annealing of a cold rolled ferritic-austenitic duplex stainless steel. *ISIJ International*, 1998, vol. 38, pp. 567-71.

[6] SOLOMON, H. D. Age hardening in a duplex stainless steel. *Journal of Heat Treating*, 1983, vol. 3, pp. 3-15.

[7] UNNIKRISHNAN, K., MALLIK, A. K. Aging behaviour of a duplex stainless steel. Materials *Science and Engineering*, 1987, vol. 95, no. 11, pp. 259-265.

[8] LOPEZ, N., CID, M., PUIGGALI, M. Influence of σ-phase on mechanical properties and corrosion resistance of duplex stainless steels. *Corrosion Science*, 1999, vol. 41, no. 8, pp. 1615-1631.

[9] KOUSSY, M. R., MAHALLAWI, I. S., KHALIFA, W., DAWOOD, M. M., BUECKINS, M., Effects of thermal aging on microstructure and mechanical properties of duplex stainless steel weldments. *Materials Science and Technology*, 2004, vol. 20, pp. 375-381.

[10] PODANY, P., DLOUHY, J., KÖVER, M., Effect of ageing on phase evolution and precipitation behaviour of duplex steel. In: *The 4th Global Conference on Materials Science and Engineering.* Bristol, MST, 2015. pp. 7-12.

[11] HUANG, C.S., SHIH, C.C. Effects of nitrogen and high temperature aging on σ phase precipitation of duplex stainless steel. *Material Science and Engineering*, 2005, vol. 402, no. 8, pp. 66-75.

[12] PODANÝ, P., MARTÍNEK, P., NACHÁZEL, J., BALCAR, M. Heat Treatment of Reactor Vessel Steel 08CH18N10T. In *AIST Steel Properties and Applications Conference Proceedings - Combined with MS and T'12, Materials Science and Technology.* Pittsburgh, MST, 2012. pp. 8-13.

[13] BALCAR, M., SOCHOR, L., NOVÁK, J., MARTÍNEK, L., FILA, P., SVATOŇ, J., TURECKÝ, V., MARTÍNEK, P., PODANÝ, P., BAŽAN J. Production and Heat Treatment of Duplex Stainless Steel Forgings. In *METAL 2012: 21st International Conference on metallurgy and Materials.* Ostrava: TANGER, 2012, pp. 66-74.

[14] SIEURIN, H., SANDSTRÖM, R. Sigma phase precipitation in duplex stainless steel 2205. *Material Science and Engineering*, 2007, vol. 444, no. 1, pp. 271-276.

[15] FARGAS, G., ANGLADA, M., MATEO, A. Effect of the annealing temperature on the mechanical properties, formability and corrosion resistance of hot-rolled duplex stainless steel. *Journal of Materials Processing Technol.,* 2009, vol. 209, pp. 770-1782.