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Microstructural Characteristics of Nickel Alloy Grade 600 After High-Temperature Thermal Cycle

The article was a distinguished paper presented at a Symposium of Welding Engineering Institutes and Departments in Istebna on 13-14.06.2017

Abstract: Alloy grade 600 is characterised by high oxidation resistance at high temperature and resistance to stress corrosion. Because of the above-named characteristics, the alloy is widely used in the chemical and food industries as well as in nuclear engineering. However, the alloy belongs to the group of hard-to-weld materials and, because of that fact, has a wide range of a solidification point, which extends the size of the liquid-sensitive fracture area extending beyond the weld pool and occurring in the partially melted zone. The susceptibility of alloys to solidification cracking is determined using high-temperature simulation. The study presents results of tests performed using a Gleeble 3800 simulator. The tests were performed to identify parameters characterising properties of alloy Inconel 600 at high temperature, during heating and cooling, i.e. nil ductility temperature (NDT), nil strength temperature (NST) and ductility recovery temperature (DRT). The identification of the above-named temperatures enabled the determination of the high-temperature brittleness range (HTBR). The material structure in the specimen rupture area was subjected to observation. The specimen fractures were subjected to observation aimed to reveal features revealing fracture types.

Keywords: alloy 600, high temperature brittleness range, hot cracks, microstructure

DOI: 10.17729/ebis.2017.6/7

Introduction

Because of its significant resistance to high-temperature oxidation and stress corrosion, nickel alloy grade 600 is widely used in the food and chemical industries as well as in nuclear power engineering [1]. However, the material proves problematic during welding as it possesses a wide range of solidification temperatures, thus increasing the liquid-sensitive fracture area extending beyond the weld pool and located in the partially melted zone [1]. The process of high-temperature simulation makes it possible to determine the susceptibility of the alloy to hot crack formation. The simulation enables the identification of parameters characterising the mechanical properties of the alloy at high temperature, during heating and cooling, i.e. the temperature at which

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the heated alloy reaches its nil strength (i.e. NST – *Nil Strength Temperature*), the temperature at which the alloy entirely loses its ductility (i.e. NDT – *Nil Ductility Temperature*) and the temperature at which the alloy regains its ductility when subjected to cooling after previous heating (i.e. DRT – *Ductility Recovery Temperature*) [2]. The determination of the above-named parameters makes it possible to identify the range of high-temperature brittleness (HTBR) as well as to determine the susceptibility of the alloy to hot crack formation [3].

Testing Methodology

The tests involved specimens having a dimeter of 6 mm and lengths of 90 mm and 116 mm. The specimens were made of nickel alloy 600, the chemical composition of which is presented is presented in Table 1. The process of high-temperature simulation was performed using a Gleeble 3800 device. The specimen was heated to a temperature below solidus, next subjected to deformation ending up in the rupture, afterwards heated to a temperature below liquidus, cooled at a preset rate to a temperature below solidus and next subjected to deformation leading to the rupture.

After a high-temperature thermal cycle, the fractures were subjected to fractographic tests using a scanning microscope. Light microscopic tests also involved the microstructure at a depth of approximately 3 mm in the specimen rupture area. The metallographic specimens were subjected to grinding, polishing and etching.

Analysis of Tests Results

The determination of the nil strength temperature (NST) involved the making of 8 specimens. Initially, the specimens were heated at a rate of 20°C/s up to a temperature of 1000°C, next at a rate of 1°C/s up to the temperature, at which the specimen ruptured. The average NST value amounted to **1377°C**.

The macroscopic analysis of the fracture obtained in relation to the NST did not reveal the specimen area reduction. The fractographic tests revealed the presence of brittle fracture. The cracking resulted from the partial melting of grain boundaries (manifested by the characteristic morphology of grain surface – see Fig. 1). The light microscopic examination of the microstructure revealed the presence of secondary fractures located between and across grains (Fig. 2).

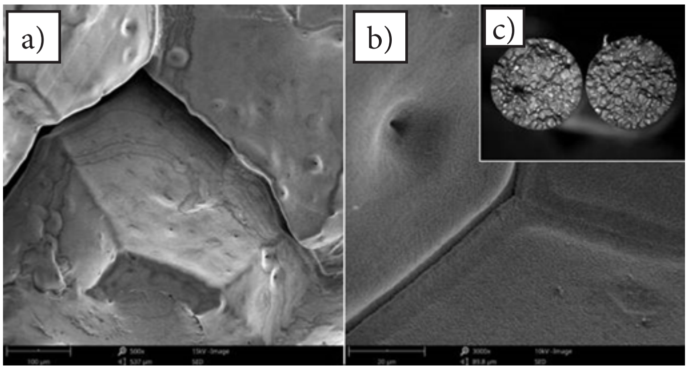

Fig. 1. Fractographic tests in relation to the nil strength temperature (NST): a) brittle fracture, b) partially melted grain boundaries, c) fracture in the macroscale

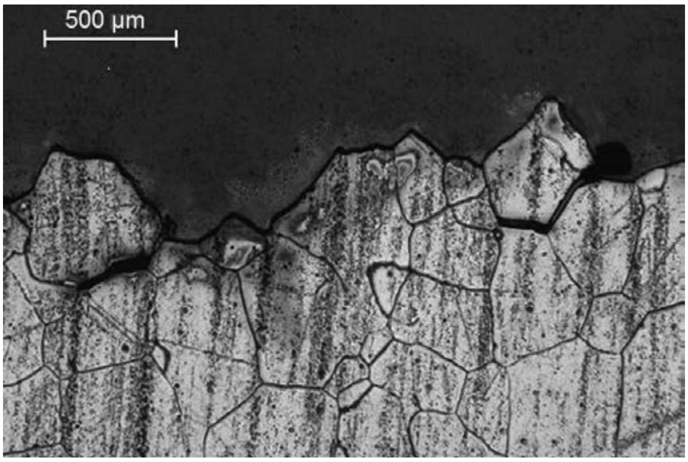

Fig. 2. Microstructure of alloy 600 in the specimen rupture area along with visible secondary fractures; etchant: 10% CrO_3; mag. 50x

Table 1. Chemical composition of nickel alloy 600

Grade	Chemical composition, % by weight							
alloy 600	Ni+Co	Cr	Fe	C	Mn	S	Si	Cu
	Min 72.0	14.0-17.0	6.0-10.0	0.15max	1.0max	0.015max	0.50max	0.50max

The determination of the nil ductility temperature (NDT) involved the making of 4 specimens. One of the results was rejected because of a measurement error. Initially, the specimens were heated at a rate of 20°C/s up to a temperature of 1250°C, afterwards at a rate of 1°C/s up to 1320°C [2, 3]. After a 5 second-long hold at the aforesaid temperature, the specimens were subjected to stress leading to the rupture. The specimen area reduction in the function of temperature is presented in Figure 3. The nil ductility temperature (NDT) was determined for an approximate area reduction of 5%. The value obtained in the tests, i.e. $Z = 7.3\%$ justified an estimation that the NDT was restricted within the range of 1320 – 1325°C.

The macroscopic analysis of the fracture revealed the specimen area reduction, i.e. strain resulting from tensile stress and the loss of cohesion in the specimen axis. The fractographic tests related to the NDT revealed a fracture of mixed nature (Fig. 4). The microstructural observations revealed numerous cracks located along grain boundaries and across grains (Fig. 5).

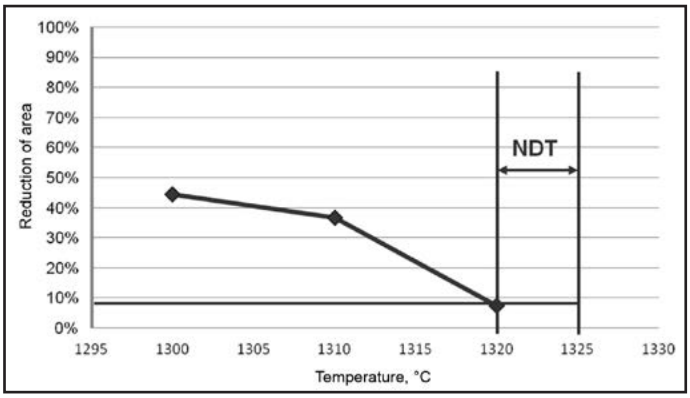

Fig. 3. Reduction of area in the function of deformation temperature in relation to alloy 600 – determination of the NDT

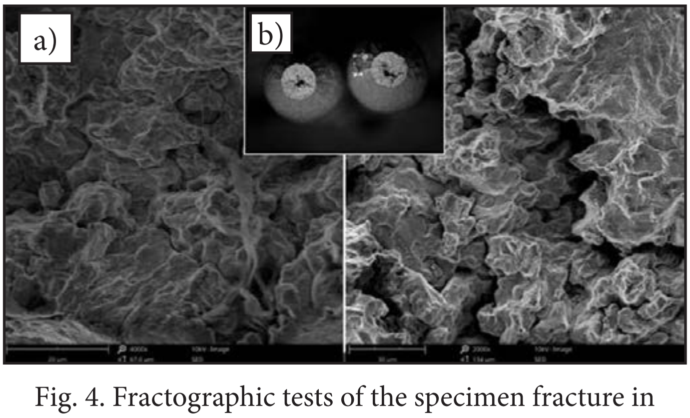

Fig. 4. Fractographic tests of the specimen fracture in relation to the nil ductility temperature (NDT): a) mixed fracture, b) fracture in the macroscale with visible specimen area reduction

The determination of the ductility recovery temperature (DRT) involved the making of 6 specimens. Initially, the specimens were heated at a rate of 20°C/s up to a temperature of 1200°C, afterwards at a rate of 1°C/s up to 1360°C [2,3]. After a 5 second-long hold, the specimen was cooled to a temperature, at which the specimen ruptured. The ductility recovery temperature (DRT) was determined when reaching an area reduction of 5%. By obtaining the value $Z = 4.66\%$ the tests enabled the determination of ductility recovery temperature DRT = 1325°C. Figure 6 presents the diagram depicting the correlation between the area reduction and strain temperature.

Related fractographic tests involved two specimens varying in temperature, at which the rupture occurred during cooling. The specimen, in relation to which the deformation temperature amounted to 1340°C revealed the presence of brittle fracture (Fig. 7). The specimen, in relation to which the deformation temperature amounted to 1320°C revealed the presence of mixed fracture (Fig. 8) and significant area reduction in the specimen axis (resulting from the recovery of ductility at the temperature).

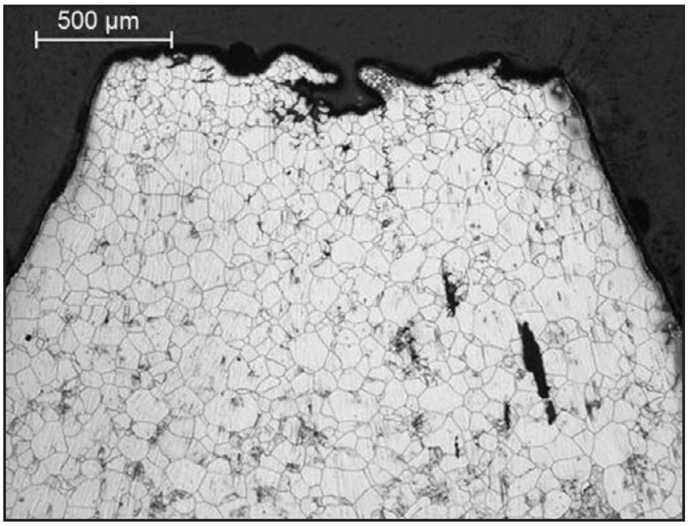

Fig. 5. Microstructure of alloy 600 in the specimen rupture area in relation to the NDT determination; etchant: 10% CrO_3; mag. 50x

The microstructural observation of the specimens used when determining the ductility recovery temperature revealed spaced grain boundaries (Fig. 9a) and clearly visible cracks located along grain boundaries and across grains (Fig. 9b).

The high-temperature simulation results were used to identify the high-temperature brittleness range (HTBR) for alloy 600 and calculate the value of crack resistance coefficient R_f. Related results are presented in Table 2.

Conclusions

The above-presented tests justified the formulation of the following conclusions:

1. The observations concerning the NDT-related specimen fracture revealed that the fracture was of mixed nature. The fractographic examinations concerning the NST-related specimen fracture revealed its intercrystalline character. The specimens used when determining DRT revealed brittle fracture in terms of the specimen for which the deformation point amounted to 1340°C and mixed fracture as regards the specimen for which the deformation point amounted to 1320°C. The foregoing resulted from the recovery of ductility at the above-named temperature.
2. The microstructure of each specimen revealed the presence of numerous secondary cracks located both between and across grains (inter and transcrystalline cracks).
3. In terms of alloy 600, the HTBR was restricted within 1377°C and 1325°C.

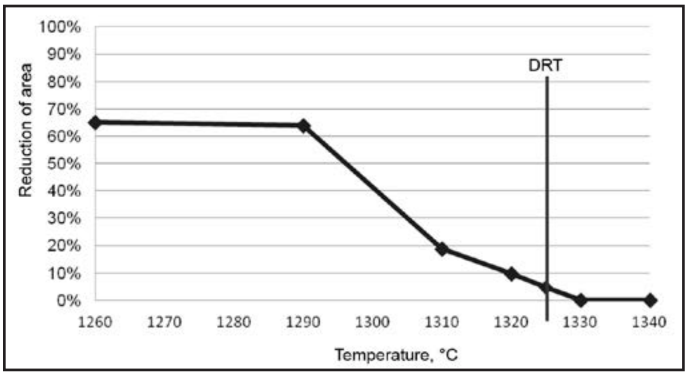

Fig. 6. Diagram of specimen area reduction in the function of strain temperature – in relation to the ductility recovery temperature (DRT)

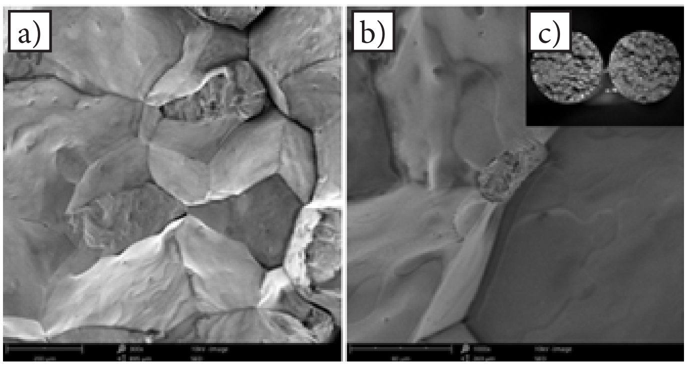

Fig. 7. Fractography of the specimen fracture in relation to the ductility recovery temperature at a strain temperature of 1340°C: a) brittle fracture with the visible partial melting of grain boundaries, b) characteristic bridge between grains, c) fracture in the macroscale with the visible lack of specimen area reduction

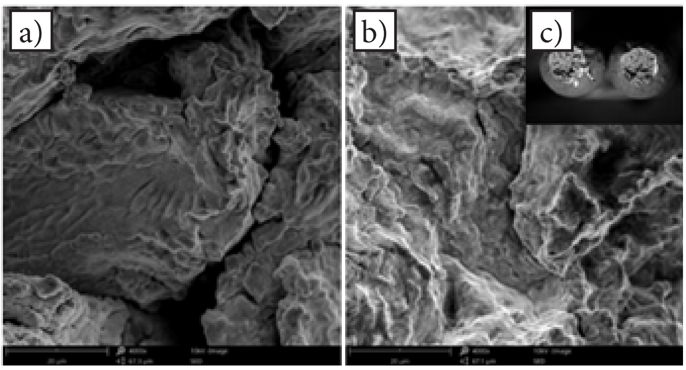

Fig. 8. Fractography of the specimen fracture in relation to the ductility recovery temperature at a strain temperature of 1320°C: a) mixed fracture, b) fracture in the macroscale with the visible area reduction in the specimen axis

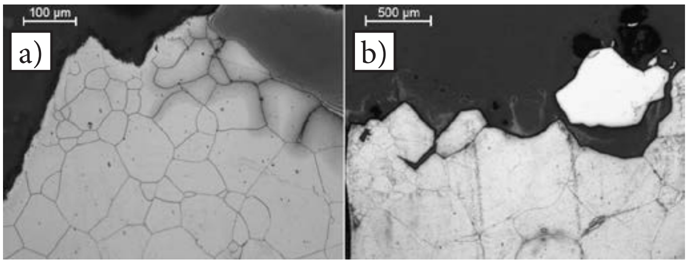

Fig. 9. Microstructure of alloy 600 in the DRT specimen rupture area along with visibly extended grain boundaries: a) mixed fracture specimen, b) brittle fracture specimen; etchant 10% CrO_3; mag. 200x

Table 2. Test results obtained in the high-temperature simulation in relation to alloy 600

	NST, °C	NDT, °C	DRT, °C	R_f	HTBR	Width of HTBR, °C
alloy 600	1377	1320 ÷ 1325	1325	0.04	1377 ÷ 1325	52

4. The coefficient identifying hot crack resistance R_f=0.04 indicated that alloy 600 was characterised by low susceptibility to solidification crack formation.

The research was financed within a Dean's grant in 2016.

References

[1] Tasak E.: *Metalurgia spawania*. Wydawnictwo JAK, Kraków, 2008

[2] Zalecki W., Łapczyński Z., Rońda J., Gnot A.: *Charakterystyki wysokotemperaturowe stopów Inconel 625 i 718*. Prace IMŻ 3, 2013

[3] Adamiec J.: *The properties of nickel alloys within the Hot Temperature Brittle Range (HTBR)* *http://www.scienceknowconferences.com*