ARCHIVES
of
FOUNDRY ENGINEERING

Published quarterly as the organ of the Foundry Commission of the Polish Academy of Sciences

ISSN (1897-3310)
Volume 15
Special Issue
3/2015

25 – 28

5/3

Degradation of Creep Resistant Ni - alloy During Aging at Elevated Temperature Part I:Mechanical Properties

M. Kaczorowski*, P. Skoczylas, Zb. Gulbinowicz
Institute of Mechanics and Printing, Faculty of Production Engineering, Warsaw University of Technology,
ul. Narbutta 85, 02-524 Warszawa, POLAND
*Corresponding author. E-mail address: m.kaczorowski@wip.pw.edu.pl

Received 18.05.2015; accepted in revised form 20.07.2015

Abstract

The results of experimental study of mechanical properties degradation of creep resistant Ni-base alloy are presented. The material studied was subjected to long-term influence of high pressure hydrogen atmosphere at temperature 750K and 850K. The mechanical properties of specimens taken at different distance from inner surface of thick wall tube were evaluated. The results of mechanical testing showed distinct influence of the temperature on mechanical properties of Ni creep resistant alloy. Moreover, the mechanical properties of studied alloy depend on the location of the specimen with respect to the dissociated ammonia source i.e. distance from the inner surface of the chamber. The results of mechanical testing show that the higher the distance from inner surface of the chamber the better mechanical properties of studied Ni base super-alloy.

Keywords: Creep-resistant Ni alloy, Mechanical properties, Hydrogen embrittlement

1. Introduction

The nickel base creep-resistant alloys invented over the past 100 years named superalloys are unique among any other heat-resistant alloys. Their outstanding creep-resistance teaching even 0,8 of their solidus temperature follows from adoption natural microstructure consisting relative stable FCC Ni-base matrix strengthened with nano-size high density particles. Contrary to Fe and Ti alloys Ni-Cr matrix is free of allotropic transformations assuring its stable structure and properties. This Ni matrix allows precipitation of relative ductile γ' instead other brittle phase like: σ or Laves phases. Moreover, Ni-Cr matrix make possible to dissolve substantial amount of alloying elements in solid solution, which precipitate during aging leading to strengthening of the alloy. The beneficial characteristic of this matrix is forming of the thin adherent oxide films protecting against ingress of harmful elements like oxygen and sulfur and outward loss of alloying elements. The Ni-Cr matrix exhibit a proclivity to homogenous nucleation of coherent ultra-fine ductile intermetallic phases of size 20-300nm. Face-centered-cubic (FCC) crystal lattice assures multiple slip systems favoring good ductility and formability. The most effective strengthening particles in Ni-base superalloys are γ' and γ'' phases. The first one are coherent precipitates with $L1_2$-type ordered crystal lattice, while γ'' precipitates appears in high Nb content nickel alloys and posses DO_{22} type body-centered tetragonal rather than FCC lattice. These nucleate homogenously and are disc-shaped particles of 30nm diameter and 5nm thickness providing higher strength than γ' phase at the same volume percent. However, according to many researchers nickel-base alloys are susceptible to hydrogen embrittlement [2-5].

Embrittlement is phenmenon that caused loss of ductility of material, which in turn make it brittle. There are a number of different forms including [9-10]:

- Environmentally Induced Carcking (EIC),
- Stress Corrosion Cracking (SCC),
- Hydrogen Embrittlement (HE),
- Corrosion

Amoung them, hydogen embrittlement is responsible for surrprising number of delayed failures and problems with the product, especially if they undergo secondary processing operations. Hydrogen embrittlement is also known as hydrogen induced cracking or hydrogen attack. Many metals and alloys such as high strength steel, titanium and aluminium alloys and many other. Nickel base alloys and creep resistant Ni alloys known as superalloys are not excludeted.

It is generally agreed that hydrogen, in atomic form, enter and diffuse through a metal surface whether at elevated or ambient temperatures. Once absorbed, dissolved hydrogen may be present ether as atomic or molecular hydrogen or in combined molecular form. Since the molecules are too large to diffuse through metal matrix, pressure builds at crystallographic deffects such like: dislocations and vacancies or discontinuities (voids, inclusion/matrix interfaces) causing minute cracks formation. Whether this absorbed hydrogen causes cracking or appear not as a complex interaction of material strength, external stresses and temperature.

Sources of hydrogen include heat treating atmospheres, breakdown of organic lubricants, the working environment, arc welding, dissociation of high pressure hydrogen gas and even grinding in wet environment. Moreover, parts that undergo electrochemical surface treatment, such as etching, pickling, phosphate coating, corrosion removal, paint stripping and electroplating are especiallz susceptible.

Although the precise mechanism is the subject of active studies, the reality is that components fail due to this phenomenon. Example of hydrogen damage include folloving:

a. Internal cracking or blistering,
b. Loss ductility,
c. High temperature hydrogen attact.

Since the metallurgical interaction occurs between atomic hydrogen and crystallographic structure of materials, the ability of material to deform or stretch under stress is inhibited. This is why it becomes brittle under stress or load. As a result, the metal or alloy will break or fracture at much lower stress than anticipated. It is this lower breaking strength that makes hydrogen embrittlement so detrimental.

The aim of this paper is to show the results of the study the mechanical properties and structure degradation of Ni-base superalloy during long-term influence of high pressure gaseous hydrogen at elevated temperature. Since the limited volume of the articles published in Archives of Foundry Engineering the results of studies were divided into two parts: part I – Mechanical studies and part II – Structure investigations.

2. Experimental procedure

The experiment was carried out in chamber in shape of thick wall tube with wall thickness g = 20mm made from commercial heat-resistant Ni alloy. The chamber was filled with dissociated ammonia under pressure of about 400 MPa. The parameters of experiment were as follow: temperature - 750K and 850K and time of about $5,4\cdot10^6$s.

The specimens for the study were cut from the thick wall at different distance from the inner surface of the chamber using EDM technique according to the scheme showed in fig. 1. The axes of the specimens were oriented parallel to the chamber axis and located at the distance from inner chamber surface x_i = 3,1 9,7 and 16,3mm, where subscript i = 1, 2 and 3.

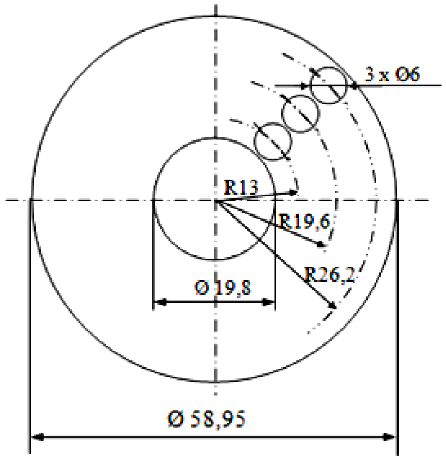


Fig. 1. Scheme illustrating from where the specimens for study were taken

At least three specimens were prepared for tensile testing and impact strength measurements. The tensile testing was performed on mini-samples 3mm diameter and 20mm length while for impact testing the smooth specimens in shape of rod 8mm diameter and 100 long were used. The tensile test were carried out on Instron 1115 with speed 1mm/min. Impact strength was measured using Charpy method with of incipient energy 150 J. For hardness evaluation Vickers and Rockwell methods were applied, and each sample was given to five measurements.

3. Results

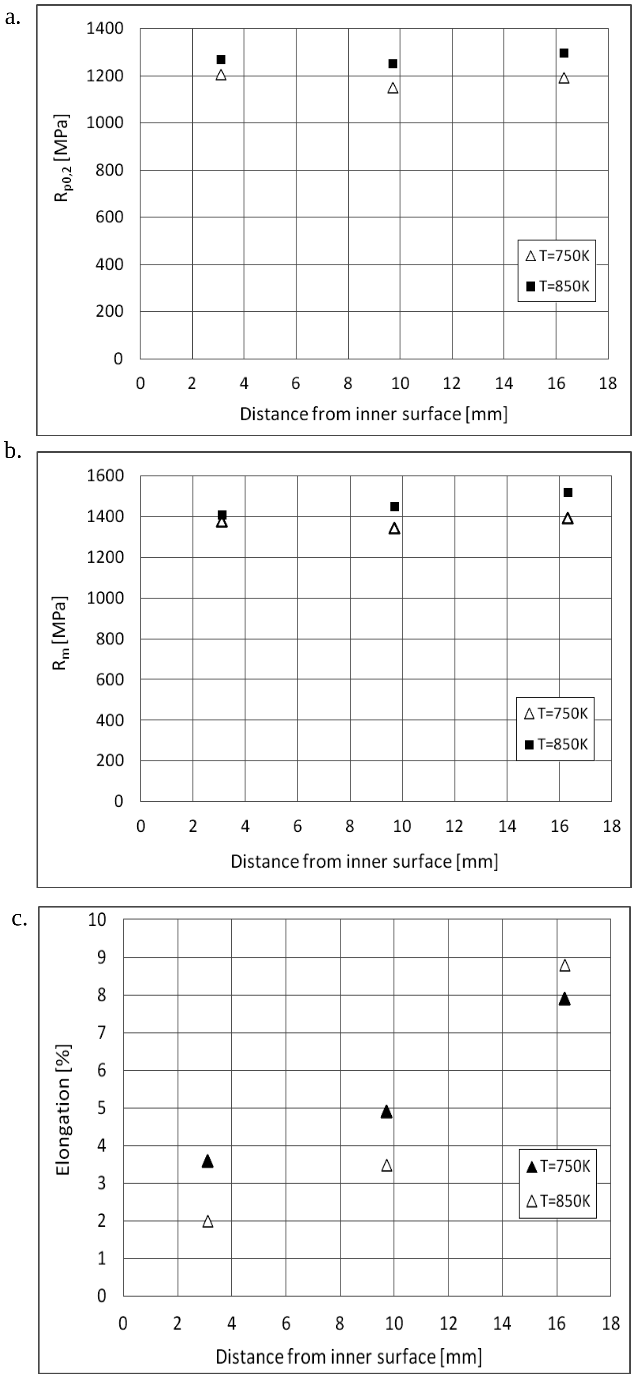
The results of mechanical testing are collected in table 1 and its graphic illustration was given below in fig. 2. It looks from very brief look on the course of the graphs that all mechanical properties of studied alloy depend more or less on temperature and the place from where the specimens were taken. If consider the proof stress (fig. 2a) the courses of $R_{p\ 0,\ 2}$ values are very similar for 750K and 850K temperatures. However, the values of proof stress in case of 750K temperature are lower than these obtained for specimen aged at 850K.

Table 1.
The results of mechanical testing

Temperature 750K						
x_i	$R_{p\ 0,2}$	R_m	A	U	Hardness	
[mm]	[MPa]	[MPa]	[%]	[J/cm^2]	HV30	HRC
3,1	1213	1371	3,5	279		
	1224	1384	3,1	278		
	1182	1377	4,3	283		
Average	**1206**	**1377**	**3,6**	**280**	**581,2**	**48,3**
9,7	1160	1346	5,2	331		
	1176	1351	4,0	313		
	1119	1341	5,4	302		
Average	**1152**	**1346**	**4,9**	**315**	**469,0**	**48,3**
16,3	1196	1398	8,8	355		
	1223	1392	8,3	322		
	1162	1393	6,7	323		
Average	**1194**	**1394**	**7,9**	**333**	**564,8**	**48,6**
Temperature 850K						
x_i	$R_{p\ 0,2}$	R_m	A	U	Hardness	
[mm]	[MPa]	[MPa]	[%]	[J/cm^2]	HV30	HRC
3,1	1271	1394	1,6	243		
	1298	1391	1,7	244		
	1237	1494	2,8	244		
Average	**1296**	**1410**	**2,0**	**244**	**578,4**	**49,4**
9,7	1263	1439	3,2	289		
	1274	1438	2,7	252		
	1221	1473	4,7	259		
Average	**1253**	**1450**	**3,5**	**267**	**573,0**	**49,2**
16,3	1301	1507	12,0	317		
	1316	1519	7,4	308		
	1271	1537	7,1	304		
Average	**1296**	**1521**	**8,8**	**310**	**562,2**	**49,4**

As follows from fig. 2b, the course of tensile strength values as a function of distance from inner surface for specimens aged at 750K is very similar in shape with that observed for proof stress showed in fig. 2a. In opposite, in case of specimen aged at 850K a little increase of R_m value with increase of distance from inner surface is observed (fig. 2b). However the most pronounced dependence of mechanical properties with distance from hydrogen source (inner chamber surface) is observed in case of elongation which increases dramatically with distance increase (fig. 2c) for temperature both 750K and 850K. Similar tendency shows impact resistance (fig. 2d) although, because of large axis scale it is not so pronounced like in case of elongation (fig. 2c). The hardness measurements carried out with HV30 method really showed minor dependence of hardness with distance (fig. 2e). The same results were obtained in case of HRC measurements which were not presented here.

Summarizing the results of mechanical investigations of nickel-base creep-resistant alloy subjected the influence of high pressure hydrogen atmosphere at elevated temperature it can be stated that it's mechanical properties degradation are dependent either on temperature and the distance from the inner surface, where hydrogen are introduced and diffuse into material.



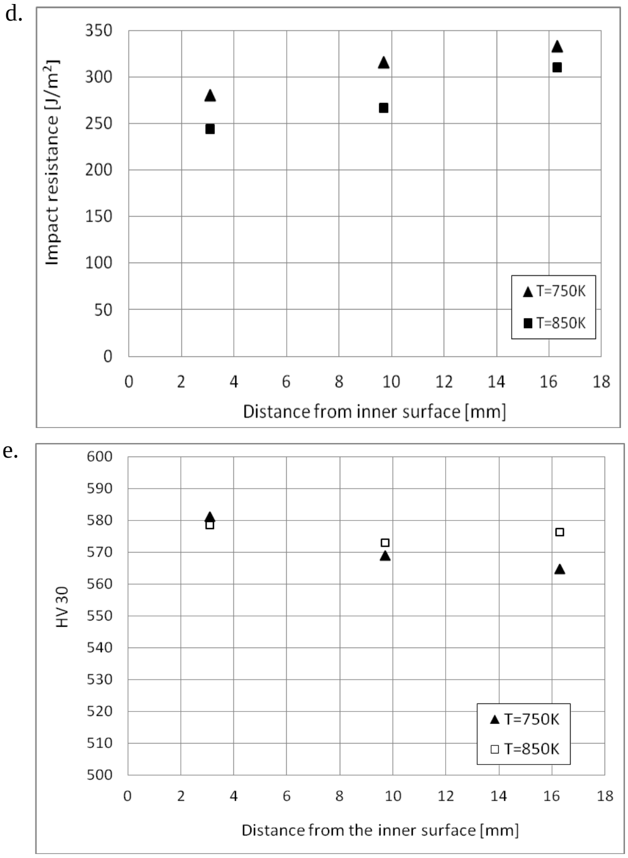


Fig. 2. The influence of distance from inner surface and temperature on: a – $R_{p\,0,2}$, b – R_m, c – elongation, d – impact resistance and e - hardness

The most intriguing looks to be a local small minimum of proof stress value observed at the middle distance from the inner surface visible in fig. 2a. Similar behavior can be identified for tensile strength of alloy heated at temperature 750K (fig. 2b)

4. Concluding remarks

Since this is the first part of the whole studies concerning the investigations of degradation of Ni-base creep resistant alloy, here we summarize some selected results only. There is no question that temperature has a substantial influence on mechanical properties of superalloy, because it determines both hydrogen diffusivity and probably the structure changes, results of which are given in the next part of this paper. The mechanical properties depend also on the depth with respect to the inner surface. It follows from the results showed above, that most pronounced changes of mechanical properties are observed for elongation and impact resistance (fig. 2d and e), where the last one is combined function of ductility and tensile strength. The hardness tests evidenced relative small decrease with x_i value increase. Such behavior is consistent with the results of elongation measurements because usually strength properties increase is accompanied with ductility decrease. To answer question what caused the changes of mechanical properties during long-term annealing at elevated temperature in hydrogen atmosphere need insight into structure of material being studied. The results of structure observation are given in part II.

Acknowledgments

This work was supported under National Center for Research and Development (NCBiR) grant INNOTECH No: K2/IN/27/27182101/NCBR/13

References

[1] Decker, R.F. (2006). *The evolution of wrought age-hardenable superalloys,* JOM, Sept., 32-38.

[2] Harris, J.A. Jr. and Van Wanderham, M.C. (1974). *Various mechanical tests used to determine susceptibility of metals to high pressure hydrogen*, in Hydrogen Embrittlement Testing ASTM STP 543, ASTM. 198-220.

[3] Harris, J.A. Jr and Van Wanderham, M.C. (1973). *Properties of materials in high pressure hydrogen at cryogenic, room and elevated temperatures*, NASA CR – 124394.

[4] Hicks, P.D. & Altstetter, C.J. (1990*)*. Internal hydrogen effects on tensile properties of iron- and nickel base superalloys, *Met. Trans. 21A.* 365-372.

[5] Hicks, P.D. & Altstetter, C.J. (1992). Hydrogen enhanced cracking of superalloys, *Met. Trans. 23A*. 237-249.

[6] Liu L., Tanaka, K., Hirose, A. & Kobayashi, K.F. (2002). Effects of precipitation phases on hydrogen embrittlement sensitivity of Inconel 718, *Sci. Technology of Advanced Materials, 3.* 335-344.

[7] He, J., Fukuyama, S., Yokogawa, K. & Kimura, A. (1994). Effect of hydrogen on deformation structure of Inconel 718, *Mat. Trans., JIM 35*. 689-694.

[8] San. Marchi C, Sommerday. Technical Reference on Hydrogen Compatibility of Materials, in Nickel Alloys: Solid Solution Alloys Ni-Cr Alloys (code 5110), eds. C. San Marchi, B.P. Somerday P. B. (2012), Sandia National Laboratories.

[9] Symons, D.M. (1997*)*. Hydrogen Embrittlement of Ni-Cr-Fe Alloys, *Met. Trans.*, 28A. 655-663.

[10] Symons, D.M. (1998). The Effect of Carbide Precipitation on the Hydrogen-Enhanced Fracture Behavior of Alloy 690, *Met. Trans., 29A.* 1265-1277.