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ScienceDirect
Acta Materialia 87 (2015) 15–24
Acta MATERIALIA
www.elsevier.com/locate/actamat

Assessment of thermal embrittlement in duplex stainless steels 2003 and 2205 for nuclear power applications

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Received 30 July 2014; revised 3 December 2014; accepted 5 December 2014

Abstract—Duplex stainless steels are desirable for use in power generation systems because of their attractive combination of strength, corrosion resistance and cost. However, thermal embrittlement at intermediate homologous temperatures of ~475 °C and below, limits upper service temperatures for many applications. New lean grade duplex alloys have improved thermal stability over standard grades and potentially increase the upper service temperature or the lifetime at a given temperature for this class of material. The present work compares the thermal stability of lean grade, alloy 2003, to standard grade, alloy 2205, through a series of isothermal agings between 260 °C and 482 °C for times between 1 and 10,000 h. Aged samples were characterized by changes in microhardness and impact toughness. Additionally, atom probe tomography was performed to illustrate the evolution of the α–α' phase separation in both alloys at select conditions. Atom probe tomography confirmed that phase separation occurs via spinodal decomposition for both alloys, and identified the presence of Ni–Cu–Si–Mn–P clusters in alloy 2205, which may contribute to the embrittlement of this alloy. The impact toughness model predictions for the upper service temperature show that alloy 2003 may be viable for use in 288 °C applications for 80-year service lifetimes based on a Charpy V-notch criteria of 47 J at room temperature. In comparison, alloy 2205 should be limited to 260 °C applications for the same room temperature toughness of 47 J.

Keywords: Duplex stainless steels; 475 °C embrittlement; Spinodal decomposition; Atom probe tomography

1. Introduction

Duplex stainless steels (DSS) are a unique class of materials that possess desirable properties of both the face-centered cubic (austenitic) and body-centered cubic (ferritic) phases within their microstructures. The ferrite and austenite phases are present in roughly equal volume fractions, typically ranging from 30 to 70% ferrite. Relative to their austenitic counterparts, DSS tend to have higher strength, higher toughness, improved corrosion resistance (especially to localized corrosion) and exceptional resistance to halide stress corrosion cracking [1,2]. Additionally, their relatively low nickel content lowers the cost of these alloys and helps to ensure price stability.

DSS are widely used in chemical processing, desalination, pulp and paper, storage and transportation industries because of their high strength and good corrosion resistance [1]. Components commonly manufactured from DSS include storage tanks, pipes, pressure vessels, heat exchangers, seawater systems, rotors and structural members. However, DSS have had little application in power generation industries, in part owing to concerns with thermal embrittlement. The thermal embrittlement that limits broader applications of DSS generally occurs at temperatures between 204 °C and 538 °C, with a peak embrittlement rate near 475 °C. The low temperature tail of this embrittlement curve is often poorly defined, owing to the long aging times required to define its location, but is critical to enable the use of these steels for long-term, elevated temperature applications of interest to the nuclear power industry. Thermal embrittlement in this temperature range typically occurs owing to the precipitation of the Cr-rich α' phase in the Fe-rich α matrix. This α–α' phase separation occurs in the ferrite grains of DSS and can occur by either nucleation and growth or by spinodal decomposition, depending on the alloy composition and aging temperature [3]. The α–α' phase separation results in hardening in the ferrite phase and a loss of toughness of the bulk material.

There are a number of commercially available DSS alloys, and there is evidence that lean grades of duplex are more resistant to thermal embrittlement than standard grades [3–5]. Lean grade alloys contain lower concentrations of Cr and Ni equivalent elements (Cr, Ni, Mo, Cu, N, C) than standard grades. Small changes in alloy composition can impact the kinetics of the embrittlement reactions in the ferrite phase [3]. Alloy 2003 (UNS S32003) is a lean

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http://dx.doi.org/10.1016/j.actamat.2014.12.012
1359-6462/

grade DSS with low Cr and Ni equivalent compositions, which make it a promising candidate for elevated temperature applications. Alloy 2205 (UNS S32205/S31803) is the most widely used DSS and is characterized as a standard grade alloy. The mechanisms and rates in which phase separation occurs in DSS alloys with different compositions is the focus of this paper.

This work characterizes the thermal stability of alloy 2003 via a series of isothermal agings, and compares it with the widely used alloy 2205. Atom probe tomography (APT) is used to identify the transformation mechanism (nucleation and growth vs. spinodal composition) and to quantify the segregation of solute species to the different phases. The degradation of mechanical properties with phase separation is characterized by impact toughness and microhardness testing. These data are compiled and fit using a form of Kolmogorov–Johnson–Mehl–Avrami (KJMA) equation in order to extrapolate the mechanical properties to times and temperatures relevant to reactor plant lifetimes.

2. Experimental details

2.1. Material

DSS alloys 2003 and 2205 were procured from the same vendor in the form of 3.8-cm-thick plates. Both alloys were solution annealed above 1010 °C and water quenched. The alloy heats and compositions are provided in Table 1. Bulk compositions were provided by vendor certifications, supplemented with independent chemical analysis [6,7]. Alloy 2205 is more solute rich than alloy 2003 in most elements, with the primary difference in higher Cr, Ni, Mo and Cu concentrations. Owing to small alloying differences and similar processing, both alloy microstructures were ~50% ferrite. The phase fraction does not change significantly during aging.

2.2. Isothermal aging

Alloys 2003 and 2205 were given a series of isothermal agings in air between 260 °C and 538 °C for times between 1 and 10,000 h to study the thermal stability. The peak embrittlement rate for α–α' phase separation occurs near 475 °C. Aging at higher temperatures can lead to σ-phase formation. Material was loaded into a hot furnace and air-cooled. The test matrix for the aging conditions and how they were analyzed are summarized in Tables 2 and 3. The shaded conditions denote APT analysis. The as-received condition of both alloys was also analyzed for impact toughness and microhardness.

2.3. Impact toughness

Charpy V-notch impact specimens were machined in the transverse–short (T–S) orientation from the aged plate of both alloys. Two or three replicate tests were performed at each test temperature. The Charpy impact machine used was capable of NIST compliance up to 434 J. Data above this impact energy are provided for information only. Impact testing procedures are in accordance with Ref. [8].

2.4. Microhardness

Specimens were sectioned from isothermally aged plates, polished and etched for microhardness testing. A minimum of 10 microhardness measurements were taken in the ferrite grains of each specimen. Measurements were taken on the short–long (S–L) surface of the rolled plate, using a Vickers indenter with a 10 gf load. This small load was necessary to avoid edge effects from the grain boundaries in accordance with ASTM standards [9]. Additional measurements were taken to better characterize the uncertainty in the measurements. The sources of variability studied include specimen-to-specimen, day-to-day test performer and replicate measurement variability.

2.5. APT

APT was performed on both alloys aged at 427 °C for times of 1, 100, 1000 and 10,000 h. Specimens were fabricated from the ferrite phase of each alloy, as evidenced by scanning electron microscopy on a mechanically ground and polished surface, by a standard focused-ion-beam-(FIB)-based in situ lift-out and annular milling method [10]. APT of the resulting needle-shaped specimens was performed with a CAMECA Instruments LEAP® 4000X HR local electrode atom probe. This instrument features an energy-compensating reflectron lens for improved mass resolution. The materials were analyzed in voltage mode with a specimen temperature of 50 K, a pulse repetition rate of 200 kHz, a pulse ratio of 0.2 and an ion collection rate between 0.5 and 2% ion per field evaporation pulse. Regions that exhibited any gallium enrichment from the FIB-based specimen preparation method were excluded from further analyses. Deconvolution of the ions within overlapping isobars of different elements (e.g. Cr_{54}/Fe_{54}) was performed based on the natural abundances of the elements.

3. Analytical procedure

3.1. Impact toughness curve fitting

Curve fits to the raw impact toughness were performed to facilitate interpretation of the test data and to help normalize the scatter in the data. Data sets with clear upper shelf energies used a hyperbolic tangent fit (provided in Eq. (1)), where E is the impact energy at a given test temperature (T_{test}) and A, B, C and T_0 are fitting constants. In the absence of a defined upper shelf, the data were fit with an exponential equation form (Eq. (2)), where a_1, b_1 and x_0 are fitting constants.

Table 1. DSS alloy compositions (wt.%).

Alloy	Heat	Fe	Cr	Ni	Mo	Mn	Si	N	C	S	P	Cu	Al	Co
2003	511,794	Bal.	21.42	3.70	1.75	1.22	0.37	0.180	0.010	0.0008	0.024	0.13*	0.01*	NR
2205	827,616	Bal.	22.44	5.69	3.11	1.80	0.42	0.17	0.020	0.0004	0.028	0.43	NR	0.33

NR = not reported.

* Value from independent chemistry analysis.