Journal of Alloys and Compounds 698 (2017) 442–450

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: http://www.elsevier.com/locate/jalcom

Thermophysical properties of Ni based super alloy 617

Arun Kumar Rai*, Haraprasanna Trpathy, R.N. Hajra, S. Raju, S. Saroja [1]

Microscopy and Thermo-Physical Property Division, Metallurgy & Materials Group, Indira Gandhi Centre for Atomic Research, Kalpakkam, 603102, India

ARTICLE INFO

Article history:
Received 26 October 2016
Received in revised form
12 December 2016
Accepted 15 December 2016
Available online 18 December 2016

Keywords:
Enthalpy
Alloy
Calorimetry thermophysical properties
Thermo-calc
JMatPro

ABSTRACT

In this study thermophysical properties of alloy 617 have been measured as a function of temperature using various thermal analysis techniques and complemented by structural and microstructural characterization. The enthalpy increment (H_T-H_{298}) has been measured using static calorimetry in the temperature range of 300–1523 K. It is found that (H_T-H_{298}) increases monotonically with temperature up to about 1000 K. However, above this temperature two distinct inflections are detected at about 1030 K and 1205 K respectively. A critical comparison of these inflections with Thermo-Calc simulations and dynamic calorimetry measurements indicates that they are associated with dissolution of $M_{23}C_6$, and γ'-Ni_3Al phases respectively. The enthalpy data has been numerically fitted using non-linear regression analysis to obtain the temperature dependence of heat capacity. Further, the thermal diffusivity and thermal expansivity of alloy 617 have been measured using the laser flash method and thermo-mechanical analyzer in the temperature range of 300–1523 K. Finally, with the knowledge of heat capacity, density and thermal diffusivity data, thermal conductivity has also been estimated in the temperature range of 300–1523 K. The measured properties for alloy 617 have been compared with Thermo-Calc, JMatPro simulation and other literature data, which clearly establishes the role of precipitates dissolution. The various properties measured in the present study are original and new addition to the database for alloy 617, which can serve as new input for Thermo-calc based optimization. This paper also provides the insight into the temperature and composition dependence of thermal conductivity of alloy 617.

1. Introduction

In recent years, the development of the Ni based super alloys which can operate beyond 700 °C has got special attention due to their intrinsic properties such as good oxidation resistance, excellent hot corrosion resistance and creep-rupture properties needed for high temperature application [1,2]. One of the most promising superalloy candidates in this context is alloy 617 (Ni–22-Cr–11.7Co–8.7Mo–1Al–0.4Ti–1Fe0.06C; *wt.%*). Alloy 617 is predominantly solid solution strengthened and has a huge potential for applications in structural components of advanced ultra supercritical (AUSC) coal based power plants [3]. The performance of alloy 617 at high temperature is crucially governed by stability of different precipitates that form during fabrication process and service conditions [3–7]. In alloy 617, three different types of precipitates namely Ni_3(Al, Ti) known as γ' phase, MC {NbC, TiC or (Nb,Ti)C} and $M_{23}C_6$ (M = Cr, Fe, Mo) type carbides are reported [5,7]. It has been witnessed that the thermo-mechanical properties of alloy 617 at high temperature crucially depend on the thermal stability of these precipitates [6]. Therefore, reliable and accurate knowledge on the kinetics of precipitation and dissolution of different precipitates is very important in assessing the phase stability of alloy 617 [3–7]. In this context, several investigations have been carried out on the characterization of microstructures and mechanical properties of alloy 617 [3–7]. In literature, the maximum attention has been paid on the microstructural evolution and secondary phase precipitation under different thermo-mechanical processing conditions to achieve the desired mechanical properties required for AUSC operating conditions [3–7]. However, in recent years, with a view to increase product quality and reduce cost in industry, there is increasing interest on advanced numerical simulations methodologies which would provide a better understanding on influence of thermal evolution, stress and microstructure during welding, casting and other manufacturing processes [8]. The success of these numerical simulations depends, to a large extent on an accurate thermophysical property database

* Corresponding author.
E-mail address: arunkumar@igcar.gov.in (A.K. Rai).
[1] Professor Homi Bhabha National Institute, Department of Atomic Energy.

http://dx.doi.org/10.1016/j.jallcom.2016.12.183
0925-8388/

[8]. Whereas, the required input data are frequently non-existent or unreliable at elevated temperature for many Ni based super alloys [9,10]. In addition, thermal properties of these materials are strong functions of their initial microstructure and alloy content; therefore, these properties must be reported for different alloy compositions and initial microstructural history [11–15]. Since alloy 617 has been indigenously developed and it is observed that experimental measurements on high temperature thermo-physical property data such as specific heat, thermal expansion, density and thermal conductivity as a function of temperature for this alloy are scarce. In this context, generation of a comprehensive thermo-physical property database for alloy 617 has been taken up using thermal analysis techniques. The thermo-physical properties obtained in this study have also been correlated with its microstructure. The results obtained in the present study have been critically analyzed in the light of available literature information and Thermo-Calc, JMatPro simulation. The experimental details adopted in the present study are described below.

2. Materials and experimental methods

2.1. Preliminary characterization

Ni base super alloy 617 tube of dimensions with ID 300 and OD 320 mm has been used in the present study. The chemical composition has been analyzed using inductively coupled plasma atomic emission spectroscopy (ICPAES) and is listed in Table 1. The tube has been flattened into sheets and solution annealed at 1473 K (1200 °C) for 2 h followed by water quenching for further experiments. For thermal analysis measurements specimen were aged at 973 K (700 °C) for 1000 h in a muffle furnace under flowing high purity Ar (ultra high purity grade 5.0 with O_2 < 0.5 ppm, N2 < 5 ppm, hydrocarbons < 0.5 ppm & moisture < 1 ppm) atmosphere at 1300 mbar pressure to promote the precipitation of different phases [7]. The aged alloy has been characterized using high precision XRD on a synchrotron source with beam energy of 14 MeV and θ range: 10-60° with Δθ step of 0.01°. The synchrotron XRD has been carried out using INDUS-2 beam line at Raja Ramanna Centre for Advance Technology (RRCAT) Indore; India which is a 2.5 GeV, 300 mA synchrotron radiation source [16]. For microstructural analysis, specimens have been prepared using standard metallographic procedures. The surface has been electrochemically etched with an etchant of 10% HNO_3 solution with constant anode potential of 2 V. The scanning electron microscopy (SEM) experiment has been carried out using Philips XL 30® microscope with an energy dispersive X-ray spectroscopy (EDX) attachment for microstructure and micro chemical analysis.

2.2. Thermal analysis characterization

Enthalpy increment ($H_T - H_{298}$) as function of temperature has been measured using SETARAM multi HTC96 type static inverse drop calorimeter under isothermal condition in flowing ultra high purity Ar atmosphere in the temperature range of 473–1523 K. In inverse drop calorimetry experiment, the alloy 617 sample of mass 100 ± 5 mg has been dropped from room temperature (T_{ref} = 298.15 K) into an alumina crucible which is well-equilibrated to a desired temperature (T) of measurement with a stability of ±0.1 K. Further the temperature of the alumina bed is monitored from the time the sample is dropped into the bed until equilibration is reached. The raw signal on Y-axis consisting of relative heat flow (from hot alumna bed to dropped sample) measured in microvolts (μV) is monitored as a function of time in seconds. After subtracting the baseline, the total peak area is calculated in terms of μV × time (seconds) that is associated with each *isothermal* drop experiment. The total area is taken to be proportional to the respective enthalpy increment, ($H_T - H_{298.15}$) of the sample at each temperature. The proportionality factor or the calorimeter calibration constant $C(T)$, which is a function of temperature, is obtained at each temperature by measuring the quantum of heat flux transacted with the simultaneous drop of a known mass of a reference material namely α-alumina for which enthalpy data ($H_T - H_{298}$) is known in terms of J/g. In the present study, α-Al_2O_3 (sapphire) was used as the reference for heat flux calibration, details of the experiment are given in a previous publication and only key points are highlighted here [17]. Data collection time of about 20 min is chosen and a time gap of another 30 min is given between two successive drops for stabilization of the alumina drop bed temperature. In order to ensure reproducibility 3–4 experiments have been carried out at each temperature. The accuracy of the ($H_T - H_{298}$) data is found to be better than ±5% up to 1273 K, however above this it is found to be of the order of ±10%.

The differential scanning calorimetric (DSC) experiments were carried out using SETARAM SETSYS 1600 heat-flux type. The instrumental and experimental details have already been presented in one of our previous publications [17] and only relevant points are highlighted here. The samples of alloy 617 are machined in the form of cubes of dimension of 2.5 mm with mass of 90 mg. These are loaded into the DSC cradle and heated up to 1773 K (1500 °C) at a slow scan rate of 3 K min^{-1} under flowing ultra high pure argon atmosphere (50 ml min^{-1}). Pure aluminum, copper, gold, and pure iron standards supplied by SETARAM have been used for the temperature calibration. However, pure Ni (Aldrich Chemicals, UK) was also used as secondary calibrant, since its thermal properties matches closely with alloy 617. The accuracy of temperature measurements is estimated to be ±2 K for the temperature range below 1373 K (1100 °C), and about ±5 K at 1673 K (1400 °C) for a heating rate of 3 K min^{-1}

The temperature dependence of bulk linear thermal expansion measurements have been carried out using thermo-mechanical analyzer (TMA 16) equipment supplied by SETARAM [18]. Sample of alloy 617 in the form of small disc of diameter 10 mm and thickness 1.5 mm with mirror finish flat surfaces is loaded on LVDT sensor and temperature induced strain ($\Delta l/l_o$) is measured, where Δl is change in length and l_o is the initial length. Pure α-alumina discs with known thermal expansion values is used as calibration standard [18]. The load applied on the sample during measurements was 5 g. Two different heating/cooling rates, namely 5 and 7 K min^{-1} were employed in the present study. Two different heating rates are used to check the reproducibility of the thermal expansivity data. The entire experiment has been carried out in flowing ultra high pure Ar atmosphere at the rate of 50 ml min^{-1}.

Thermal diffusivity as a function of temperature of alloy 617 has been measured using LFA 1000 by laser flash method on 10 mm diameter and 2.5 mm thick disc shaped samples with smooth surfaces. Details about these experiments and data analysis are given in our previous paper [19]. Each sample has been coated with a thin layer of graphite to ensure maximum absorption and heated in the SiC furnace at 5 K min^{-1} to the desired temperature. The temperature stability of present equipment during isothermal holding was ±1 K. At the desired temperature, the sample has been irradiated with an Nd-YAG laser source of pulse energy 25 J/pulse with a pulse length of 300μs on its front surface and its temperature

Table 1
Chemical composition of alloy 617 used in the present study. All the composition are listed in wt.%.

Ni	C	S	Mn	Si	Cr	Co	Mo	Al	Ti	Nb	Fe	V
Bal.	0.06	0.002	0.03	0.09	21.9	11.7	8.7	1	0.4	0.07	1	0.01