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Thermal characterization of a Ni-based superalloy

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Abstract

Ni-based superalloys are usually employed in industrial applications where good mechanical performance, corrosion and oxidation resistance at high temperatures is required. Samples collected from a gas turbine rotor blade were used in the present study. The X-ray photoelectron spectroscopy (XPS) and inductive coupled plasma (ICP) techniques were used to identify and quantify the elements in the alloy. simultaneous thermal analysis (STA) has been used to study the behavior of the alloy during the heat treatment from room temperature to 1450 °C, under argon and air atmospheres. Heat capacity has been evaluated and two solid–solid transitions have been identified below 1400 °C. A small oxidative process was identified at temperatures over 1150 °C.

Keywords: Corrosion; Superalloy; Simultaneous thermal analysis

1. Introduction

Many industrial applications such as gas turbine rotor blades require materials with special properties in relation to corrosion and oxidation resistance at high temperatures and mechanical performance in a wide range of temperatures. Several commercially available of Ni-based superalloy show these promising performances. Further knowledge about the interaction between the composition and thermal treatment of the alloy will determine the future development of this alloy.

Most of these alloys depend on their oxidation and hot corrosion resistance upon chromium and on their strength upon aluminum and titanium to allow the precipitation of an ordered compound based on the formula Ni_3 (Al,Ti). This phase is called γ' to distinguish it from the face-centered cubic matrix configuration and denoted as γ [1].

Aluminum and titanium are often used in minor amounts in corrosion-resistant alloys for the purpose of deoxidization of carbon and nitrogen. Chromium additions improve resistance to high temperature oxidation and attack by hot sulfur-bearing gases [2].

Most high-strength nickel-based alloys depend on the precipitation of an A_3B-type compound known as γ'. Ni simple alloys takes on the form Ni_3(Al,Ti). In complex alloys, other elements can substitute for nickel on "A" side or for aluminum and titanium on the "B" side [3].

Microstructural changes induced by a thermal treatment have been described for some nickel-base superalloys [4]. Thermal analysis had been applied to study the hard metals [5]. Precipitation phenomena were analyzed in metal alloys by thermal analysis [6]. Oxidation of different alloys had been studied by means of TGA [7,8].

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2. Experimental

The samples were collected from a gas turbine rotor blade. X-ray photoelectron spectroscopy (XPS) and inductive coupled plasma (ICP) were used to determine the chemical composition. The carbon content was obtained from the analysis by means of a Leco CS-300 apparatus.

Samples for thermal analysis were cutted-off with a wheel to the proper size and shape and were analyzed in a Rheometric Scientific Simultaneous Thermal Analyzer STA 1500 with system interface device and a computer workstation. All the samples were placed in the alumina crucibles. Three types of experiment had been performed:

1. original sample under argon environment;
2. a second and identical treatment was applied to the same sample;
3. new original sample under dried air environment.

A heating step from 25 to 1450 °C at a heating rate of 10 °C/min, was followed by a cooling step from 1400 to 25 °C. Cooling rate was also maintained at 10 °C/min. The flow rate of the gas was kept at 50 ml/min in all the experiments.

Microscopic analysis was performed before and after the thermal analysis experiments. The samples were mounted in a phenolic resin, grinded and then polished. The samples were dipped for 10–15 s in an etching agent [9]. The etching solution composed of 92% of HCl, 5% of H_2SO_4 and 3% of HNO_3 by volume.

3. Results and discussion

Chemical composition of the alloy was Ni 69.5%, Cr 9.2%, Co 9.1%, Al 5.1%, Ti 4.5%, Mo 2.5%, Fe 0.06% and $C < 0.02$. These compositions are very common in gas turbine rotor blades.

Figs. 1 and 2 show the micrographs of the alloy obtained from scanning electron microscope (1000× magnification), before and after the thermal treatment, respectively. Those pictures display a clear difference in microstructure from the original acicular shape before the thermal treatment to a rounded granular shape after the thermal treatment. This new structure was probably developed because of the lower cooling rate compared with processing conditions.

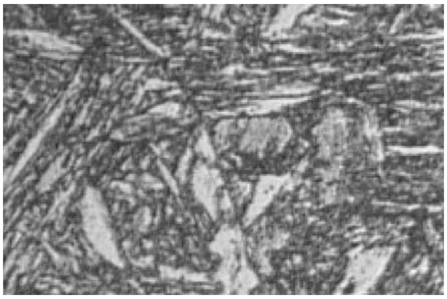

Fig. 1. SEM micrograph before the thermal treatment.

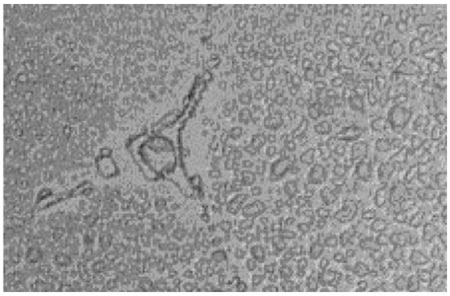

Fig. 2. SEM micrograph after one scanning on the STA.

Fig. 3 shows the DSC and TGA traces for the original sample with argon was as purge gas. The second identical treatment to the same sample as illustrated in Fig. 4. There is insignificant weight increase observed at temperatures above 1150 °C under argon environment. It is probably due to an oxidizing process ignited by the small amount of oxygen presence as impurity of the argon.

Similar increment of weight increase was observed when air was used. The oxidizing process depends very much on the temperature but not on the concentration of the oxygen. The heating step of the DSC trace in Fig. 3 shows two endothermic peaks which are probably due to the precipitation of some components. The cooling step of the same trace shows only one exothermic peak.

Fig. 4 shows only one peak in the heating step. The different phenomena shown in Figs. 3 and 4 are related to the different microstructures shown in Figs. 1 and 2.

The heat capacity calculation had been performed from the original sample thermal experiment under argon flow. The same alumina crucibles had been used to obtain the baseline and the synthetic sapphire (α-Al_2O_3) was used as specific heat standard. Values

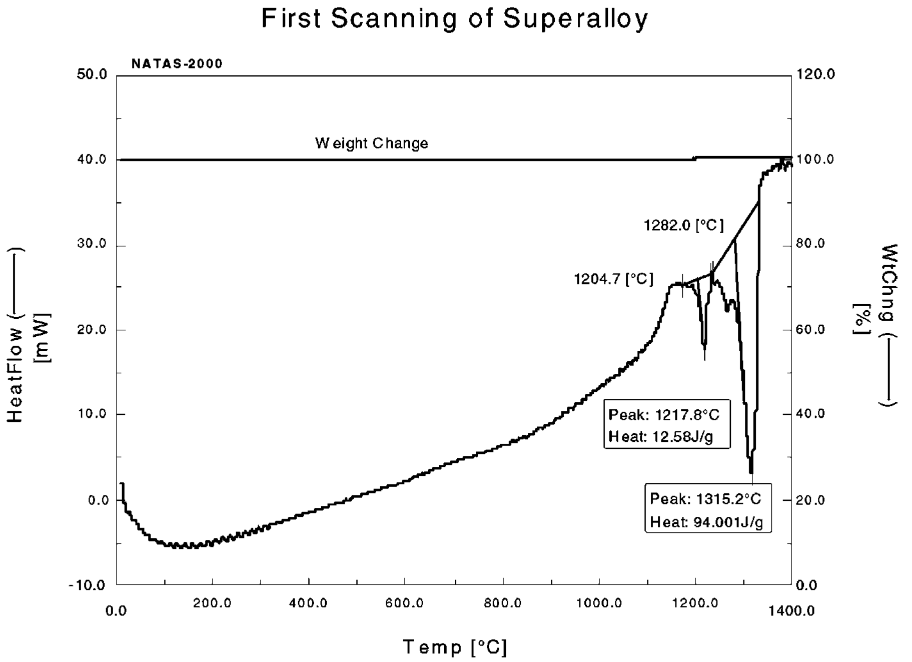

Fig. 3. The first scanning of the sample under argon.

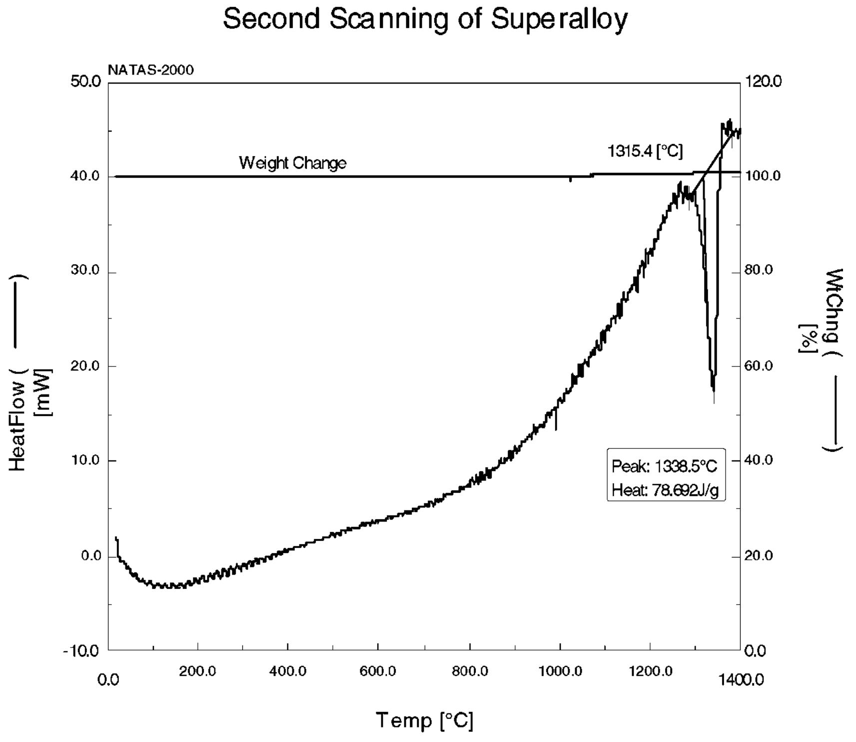

Fig. 4. The second scanning of the sample under argon.

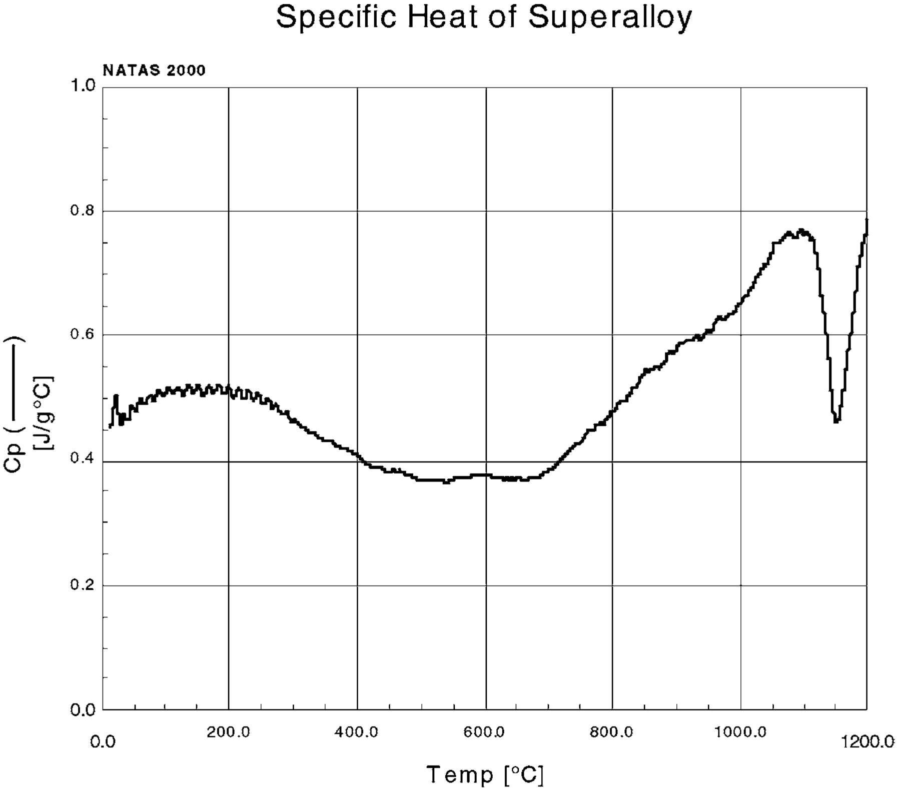

Fig. 5. Calculated specific heat for the superalloy.

of the specific heat standard were taken from the polynomial proposed by Gmelin and Sarge [10]. Fig. 5 shows the plot of the specific heat of the superalloy as a function of temperature. This plot fits well to the polynomial written in the same picture.

4. Conclusions

Samples of a Ni-based superalloys collected from a gas turbine rotor blade were analyzed. Chemical composition was determined and the microstructure was studied before and after the thermal treatment.

Simultaneous thermal analysis (STA) experiments from room temperature to 1450 °C and followed by a cooling ramp, under argon and air atmospheres, revealed transformations during the heating and the cooling steps. The changes observed in the microstructure agreed with the thermal analysis results. A small oxidative process was identified at temperatures over 1150 °C and the heat capacity of the superalloy was calculated.

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