

High Temperature Creep Testing of a Superalloy Bond Coat

Using the xSol System to Study Time-Dependent High-Temperature Deformation of Thin Layers

Introduction

Superalloys are materials developed specifically to retain high strength and resistance to creep during operation at very high temperatures. Modern superalloys are complex materials, often composed of a dozen or more constituent elements. The performance of a superalloy may be further enhanced by application of a thermal barrier coating (TBC) which is a ceramic coating that encapsulates the component and offers high thermal resistance and environmental protection. The TBC is bonded to the superalloy by means of a bond coat that is formed when the alloy is held at high temperature for an extended period of time in the presence of oxygen. During formation, oxygen diffuses inward from the surface while various constituent elements diffuse outward toward the surface at varying rates. The resulting microstructure is a complex multilayer system that provides a strong, tough bond between the metal interior of the part and the oxide TBC.

Because the mechanical properties of all materials change with temperature, measurement of the behavior of the various bond coat layers over a wide temperature range is an essential component in modelling and designing superalloys with improved properties. Because the layers are generally only microns thick, conventional means of testing the high temperature creep behavior of individual layers in isolation are difficult or impossible. Nanoindentation provides a suitable method to study the time and temperature dependent mechanical properties of the layers to understand the complex mechanical interactions that occur when superalloy components are used under service conditions.

Procedure

A sample of a commercially available CM-247LC nickel based superalloy was heated in air to produce a compositionally graded bond coat. The sample was cross-sectioned and polished to reveal the microstructural layers, and the SPM image in Figure 1 shows various microstructural zones which were distinguishable by their differing surface textures. Zone

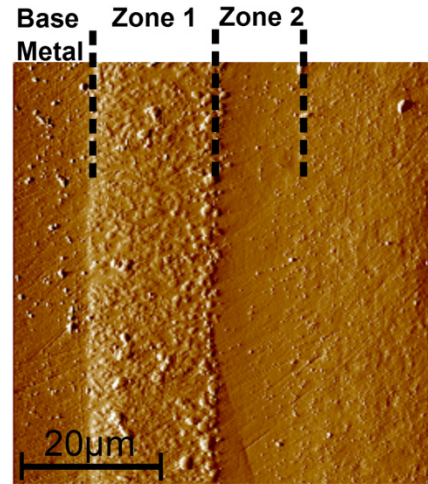


Figure 1: SPM image of the cross-sectioned sample surface showing the bond coat layers that were characterized. Images are collected with the same probe tip used to perform the tests, permitting very precise test positioning.

1 was composed of a NiAl matrix with W and Cr precipitates while Zone 2 was composed of a Pt, Ni, Al solid solution. Nanoindentation creep tests were performed on both layers over temperatures ranging from 25 to 750 °C. A dynamic creep testing technique was employed, whereby a small oscillation at a particular reference frequency (in this case 220 Hz) was superimposed onto the quasi-static load function, permitting contact stiffness to be measured continuously throughout the test. The reference creep test relies on the relationship between contact area and contact stiffness to calculate the material’s properties over long time periods. Nanoindentation creep tests lasting 1500 seconds were performed at 25, 500, 650, and 750 °C on Zone 1 and Zone 2 using a **TI 950** nanomechanical test system equipped with a Berkovich indenter probe.

Results

The locations of the nanoindentation creep tests were selected and confirmed using the instrument’s *in situ* SPM imaging capability. An example of an SPM image collected at 650 °C is given in Figure 2. Figure 3 (left) shows data from a creep test at each temperature in each zone, demonstrating how the indent depth increased over time at constant quasi-static load. Figure

3 (right) shows the corresponding decrease in hardness associated with the increasing indent depth. Tests at 25 °C were performed to serve as a baseline because very little creep was expected at room temperature. As temperature increased, the initial hardness decreased and the creep rates accelerated. Creep in the steady-state regime is described according to the equation:

$$\dot{\epsilon} = A\sigma^m e^{-Q/RT}$$

Where $\dot{\epsilon}$ is the strain rate, A is a proportionality constant, m is the stress exponent, Q is the activation energy, R is the gas constant, and T is the absolute temperature. Changes in the creep mechanism are associated with changes in the stress exponent, m , and/or the activation energy, Q . Since stiffness is known continuously during a reference creep test, the strain rate can also be calculated continuously as:

$$\dot{\epsilon} = \dot{k}/k$$

Where k is the contact stiffness. The hardness, or mean contact pressure, may be taken as the representative stress. The stress exponents can then be calculated by taking the slope of $\log \dot{\epsilon}$ vs. $\log H$, as shown in Figure 4 (left). Note that during the first 100-200 seconds of the tests, transient behavior was observed as non-linearity in the $\log \dot{\epsilon}$ vs. $\log H$ curves. Such data was omitted from Figure 4 (left) since only the steady-state behavior was of interest for this study. Figure 4 (right) shows the stress exponent as a function of temperature and suggests that a single creep mechanism dominated over the temperature range tested for Zone 1 while the large changes in stress exponent for Zone 2 suggest that there were competing mechanisms at work.

Conclusions

Nanindentation creep testing permits measurement of the creep behavior of small volumes of material, including individual thin layers in a complex multilayer system. The use of nanoDMA® III combined with the xSol High Temperature Stage on the TI 950 platform enables measurements to be precisely positioned on the layers of interest and performed over extended time durations at temperatures up to 800°C.

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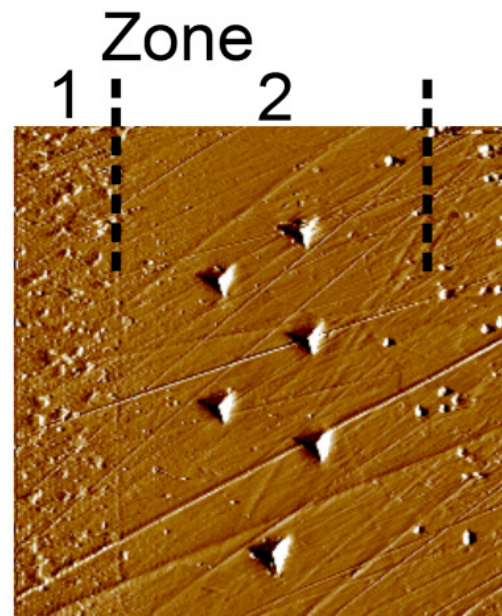


Figure 2: SPM image of indent impressions in Zone 2 collected at 650 °C.

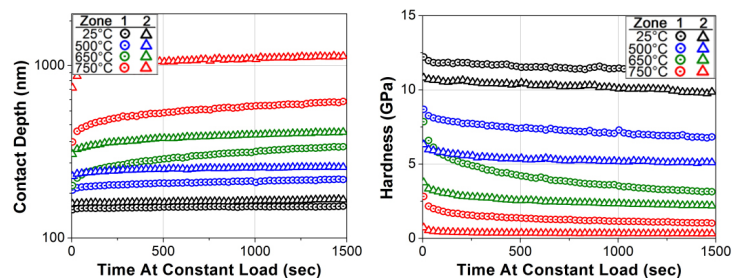


Figure 3: (Left) Creep data from each temperature showing the evolution of indent depth during the test. (Right) Decaying hardness over time at each temperature associated with increasing indent depth.

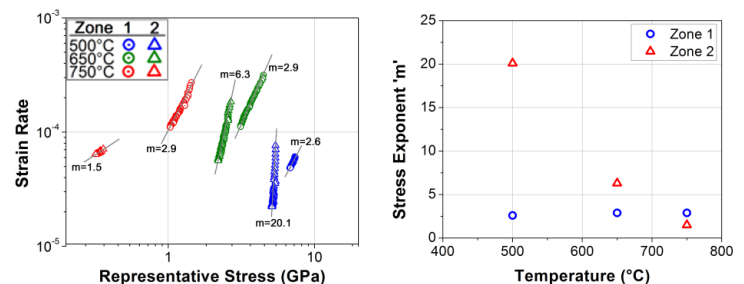


Figure 4: (Left) Strain Rate Vs. Stress from each creep test, showing how stress exponents are calculated. (Right) The changing stress exponent for Zone 2 suggests a changing creep mechanism while the consistent results for Zone 1 suggest a constant mechanism.