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Pore annihilation in a single-crystal nickel-base superalloy during hot isostatic pressing: Experiment and modelling

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ABSTRACT

Pore annihilation during hot isostatic pressing (HIP) was investigated in the single-crystal nickel-base superalloy CMSX-4 experimentally by interrupted HIP tests at 1288 °C/103 MPa. The kinetics of pore annihilation was determined by density measurement and quantitative metallography. Transmission electron microscopy of a HIPed specimen showed that the pores shrink via dislocation movement on octahedral glide planes. Theoretically pore closure under HIP condition was modelled by the finite element method using crystal plasticity and large strain theories. The modelling gives a similar kinetics of pore annihilation as observed experimentally, however somewhat higher annihilation rate.

1. Introduction

Blades of aircraft gas turbine engines and land-based power gas turbines operate under severe service conditions such as high temperature, different types of loading and the effect of aggressive burning products. Therefore the most advanced technologies are used for manufacturing of these blades. In order to exclude the intergranular oxidation and rupture, the turbine blades are solidified as single-crystals of nickel-base superalloys. Industrial manufacturing of the single-crystal blades is performed by the Bridgeman method with a temperature gradient of 5–20 °C/mm and a withdrawal rate of 3–25 mm/min depending on the cooling method [1]. Under such solidification conditions the superalloy crystal grows by dendritic growth which results in the formation of micropores between the dendritic arms. Additional porosity forms via the Kirkendall effect during the solution heat treatment. These pores are small, about 10–30 μm, and their volume fraction is just a few tenths of a percent [2,3]. However, these micropores significantly deteriorate the fatigue strength of single crystal superalloys because under cyclic loading the rupture is initiated by crack nucleation at pores [4,5]. This deteriorating effect is especially pronounced at temperatures below about 750 °C, where the material ductility is low, so that the plastic damage accumulated at pores cannot be recovered by local creep.

It was shown in [6] that microporosity in single-crystal superalloys can be removed by hot isostatic pressing (HIP) and now advanced companies apply this technique to turbine blades. However this technological process, performed at temperatures close to solidus, bears the risk of recrystallisation and incipient melting. Single-crystal blades are expensive and the price of HIP is high as well. Therefore the parameters of industrial HIP (temperature T, pressure p and duration t) have to be carefully optimised under the conditions complete annihilation of the pores, no material damage and minimal costs of processing. The HIP optimisation could be simplified, if the pore closure mechanism would have been identified. This would allow developing a numerical model for pore closure and then to study the effect of different HIP parameters on the kinetics of pore annihilation. The presented work is an attempt to solve this task.

2. Experimental

2.1. HIP experiments

The investigations were performed with the Nickel-base superalloy CMSX-4® [1] [7] widely used as blade material for gas turbines. Single-crystals of this alloy were solidified in the [001] direction by Howmet Castings, Alcoa, USA. As mentioned, the pores are partially generated during homogenisation. Homogenisation however is a side effect of HIPing and in some industries both

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[1] CMSX-4® is registered trademark of the Cannon-Muskegon Corporation.

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processes are performed in one step. Therefore one part of the single-crystals was left in as-cast condition, whereas the other one was solution heat treated. The kinetics of pore annihilation during HIP experiments was investigated by transmission electron microscopy (TEM) and the pore annihilation mechanisms were identified.

The HIP experiments were performed in the HIP plant QIH-16, ASEA at 1288 °C/103 MPa/6 h in argon, as used by Howmet Castings for CMSX-4. In order to investigate the kinetics of pore closure the HIP experiments were interrupted after 0.5, 1, 2 and 4 h, but also continued until the full HIP time of 6 h. The change of temperature and pressure during HIPing is presented in Fig. 1. It also shows that the HIP temperature is above the γ′-solvus temperature which is for superalloy CMSX-4 about 1280 °C. Such a small exceed above the γ′-solvus temperature is advantageous for HIPing because in single phase condition the superalloy is plastically much softer. The technical premise for this kind of HIPing is, that the temperature difference between chamber bottom and top is less than 5 °C. Both types of specimens, as-cast and solution heat treated ones, were HIPed. For density measurements the specimens had cylindrical shape with 18 mm diameter and 45 mm length. For quantitative metallography plates with 3 mm thickness were used.

2.2. Density measurement and quantitative metallography

The change in porosity during HIP can be calculated from the change of the density ρ. As reference material a heat treated unHIPed specimen was used. Density was measured by the Archimedes method, based on the difference of weight, when the cylinder is surrounded by air, respectively water. This technique was refined in our laboratory and a relative accuracy of $\Delta\rho/\rho \approx 10^{-4}$ was attained [8], sufficient to measure the kinetics of porosity annihilation, which covers a range of about 0.1–0.2 vol %. The ratio between specimen surface and volume was reduced by taking specimens as big as possible, in our case about 100 g. To avoid gas bubbles attaching to the specimen surface during the measurements in water, the corners of the cylinders were rounded and the surface polished very carefully. During the measurements the temperature of air and water was kept constant within ± 0.1 °C and the measurements were performed several times to check the repeatability. The used precision balance, Sartorius R160D, has an accuracy of 0.01 mg. The measured relative increase of density is equal to the decrease of the pore volume fraction. In order to know the absolute value of the pore volume fraction in the reference specimen, its porosity was quantitatively analysed in a scanning electron microscope (SEM). Twenty images were taken at a magnification of 200 and then the porosity measured as described below.

Because porosity measurement via density is not a direct method, the kinetics of pore annihilation was measured additionally by quantitative metallography. For this purpose specimens were cut, thoroughly polished and cleaned and then images taken in a Zeiss Axioscope light microscope at a magnification of 100. Fifteen to 20 images were processed with the Olympus software a4i for each porosity data point.

2.3. Transmission electron microscopy

The main aim of the TEM investigations was to find an indication of the pore annihilation mechanism near the pore surface. The investigations were performed in a Phillips CM30 TEM. The mechanism of pore annihilation was checked with a specimen, where the HIP test was interrupted after 0.5 h. During this time the pores shrink significantly but still many pores exist which did not close completely. In order to attain a TEM foil of such an unclosed pore, the specimen was cross cut and the polished surface scanned for remaining pores. In a focused ion beam (FIB) workstation FIB200 from FEI, an unclosed pore was filled by deposition of platinum in order to avoid damage during the subsequent foil preparation. Then a TEM lamella was cut perpendicular to the specimen surface by focused ion beam (see Fig. 2a). The vertical lines are artefacts from ion cutting.

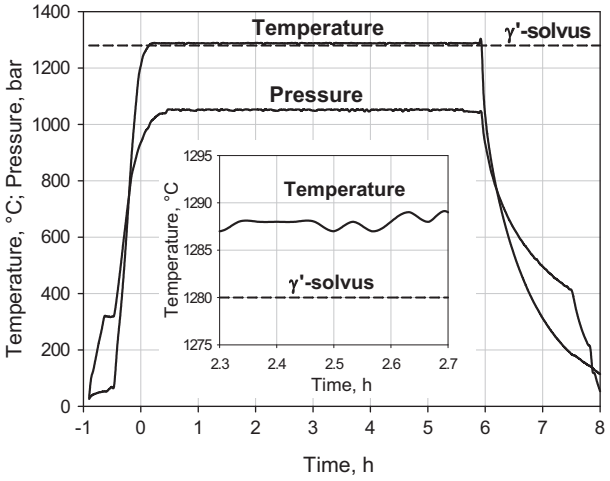

Fig. 1. Change of temperature (in bottom of chamber) and pressure during HIPing CMSX-4 in the HIP plant QIH-16, ASEA.

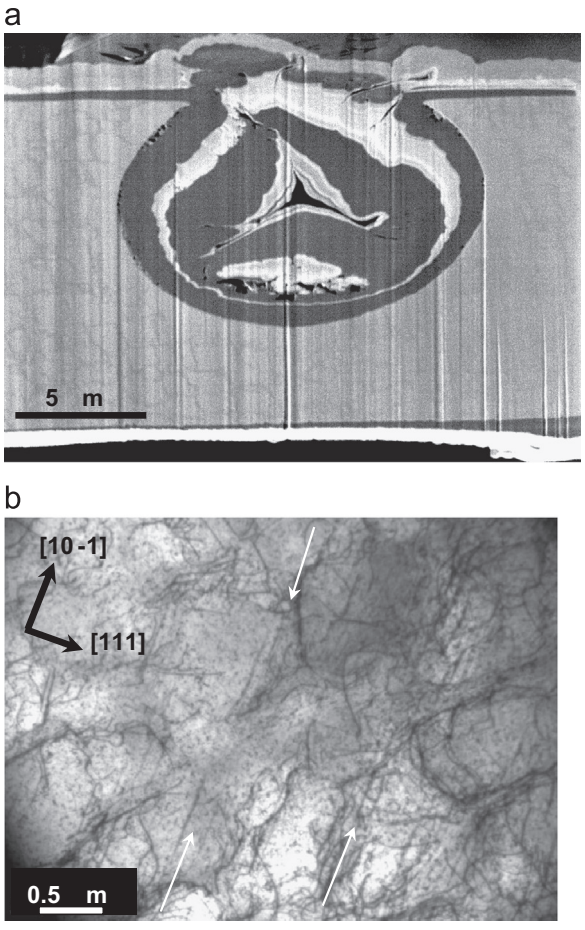

Fig. 2. Superalloy CMSX-4 after HIP at 1288 °C/103 MPa/0.5 h. (a) TEM foil cut by FIB. The pore is filled by Pt. (b) TEM image. (111) Slip planes in edge on position, marked by white arrows. $\vec{k}_0 = [1\bar{2}1]$, $\vec{g} = 111$. The distance from the pore surface is about 3 mm.