

Multiscale study of the boron and carbon behavior in the fracture zone of the rapidly quenched nickel-based superalloy by the nuclear physics methods of the track and activation autoradiography

A V Shulga

National Research Nuclear University MEPhI (Moscow Engineering Physics Institute), Kashirskoe highway 31, Moscow, 115409, Russia

E-mail: avshulga@mephi.ru

Abstract. The results of the study of the fracture zone of the rapidly quenched nickel-based superalloy after tensile tests are presented. The main attention was attended to the multiscale investigation of boron and carbon distribution in the area of the cracks propagation by the direct nuclear physics methods of the track and activation autoradiography. Tensile tests were performed at the temperatures up to 1150 °C. The nuclear reactor MEPhI IRT-2000 and cyclotron were applied for the autoradiography studies. Significant depleting of boron in the fracture zone which was revealed can be explained due to the intensive moving of the dislocation.

1. Introduction

Nickel based superalloys are the high temperature materials widely applied in critical components of a gas-turbine [1-6]. Microalloying of boron and carbon being used to improve the mechanical properties, in particular, the high temperature strength, ductility and cohesive strength of the grain boundaries [7-11]. Special attention should be given to the role of boron and carbon in the advanced rapidly quenched Ni-based superalloys manufactured by the powder metallurgy via hot isostatic pressing (PM HIP) of the melt-atomized powders. Such features of the rapidly quenched structure as high dispersivity of the microstructure elements, small dendrite arm spacing, super saturation of solid solution and the large interparticle surface in the HIPped compact provide the possibility of the essential peculiarities of boron and carbon behavior in the PM HIP Ni-based superalloys. The features of the crack propagation in Ni-based superalloys, especially in advanced rapidly quenched PM HIP superalloys have the significant meaning for estimation of their mechanical properties.

The important aim of this work is a study of the behavior of boron and carbon in fracture zone of the PM HIP Ni-based superalloy by the high sensitivity direct autoradiography technique. Simultaneously investigation of the specimen structure and the autoradiographs on boron and carbon was performed at a wide range of magnification. The technique of autoradiography allows for a multi-scale analysis of boron and carbon distribution in the various elements of the structure including macro-, metho-, and microstructure. A detailed correlation study of the structure PM HIP Ni-based superalloy compact in the fracture zone and boron, carbon distribution was performed.



2. Experimental details

The composition of the investigated PM HIP Ni-based superalloy EP741NP is listed in Table 1. The specimens with the width and thickness of 3 mm and the working length of 6 mm were cut from the as-HIPped compact of the Ni-based superalloy EP741NP.

Rapidly quenched powders were produced by plasma rotating electrode process (PREP).

Table 1. Chemical composition [in wt.%] of the PM HIP Ni-based superalloy EP741NP

Ni	Al	Ti	Cr	Co	W	Mo	Nb	Hf	C	B
Remaning 56.1	5.1	1.8	9.1	15.7	5.5	3.8	2.6	0.29	0.04	0.012

HIP parameters were the following: 1170 °C, 4 h, 150 MPa. Powder was produced by the plasma rotating electrode process (PREP). The main aim of the tensile tests was to reveal the effect of temperature on the flow behavior and mode of fracture of the PM HIP Ni-based superalloy EP741NP. The tensile tests were carried out at the temperatures 800 – 1150 °C and at 10^{-1} , 10^{-3} s $^{-1}$ strain rates.

The specimens were studied by light metallography, autoradiography on boron and carbon, Scanning Electron Microscopy (SEM), and X-ray Spectral MicroAnalysis (XRSMA). Method of the track autoradiography on boron has been provided the possibility to study macro-, metho-, and micro inhomogeneity of boron and has been used to detect the presence of boron in various elements of the structure such as solid solution in the volume of grain, segregations at the grain boundaries and dispersed boride precipitates. The autoradiography method on boron is based on the use of the nuclear reaction $^{10}\text{B}(\text{n},\alpha)^7\text{Li}$ on thermal neutrons. Fission products, mainly α -particles, were recorded by means of a plastic film detector. This nuclear method has a high sensitivity (the minimum detectable concentration of boron is of about 1 ppm) and allows investigating the multiscale spatial boron distribution with a low (macro) and a high resolution (metho- and microstructure). Dark/bright areas on the negative autoradiograph image correspond to a lower/higher concentration of tracks and therefore to the lower/higher concentration of boron. Separate tracks are detected as small black dots on the positive autoradiographs with various densities depending on boron concentration in solid solution. The coincidence of separate tracks from boron atoms in the boride precipitate results in formation of the spot on the autoradiograph which size depends on the size of boride particle.

An activation autoradiography method was used to obtain the multi-scale distribution of carbon ranging from the macro inhomogeneity in the specimen macro structure to the micro inhomogeneity in the microstructure elements. The autoradiography method is based on the use of the nuclear reaction $^{12}\text{C}(\text{d},\text{n})^{13}\text{N}$ on carbon atoms with 2 MeV deuterons. Secondary β -particles were detected on the specimen surface by the β -radiometer. This nuclear method has a high sensitivity and includes the operation of measuring the induced activity of the β -radiation due to the nuclear reaction. It must be noted that simultaneously with the more uniform distribution of carbon in the specimens, the induced activity of the β -radiation detected during the autoradiography investigation was increased.

3. Results and discussion

Figures 1 – 3 show macro- and microstructure, activation and track autoradiographs of carbon and boron distribution in the specimen of the rapidly quenched PM HIP Ni-based superalloy EP741NP after the tensile tests. Significant decreasing of boron concentration in the fracture zone of the specimen of the rapidly quenched PM HIP Ni-based superalloy EP741NP compact was observed on the track autoradiograph of boron and was confirmed by the dissolution of boride precipitates detected by scanning electron microscopy and x-ray spectral microanalysis. The length of this boron depletion area is about one millimeter from the fracture surface. Essential boron depletion in the fracture zone under testing at the temperature 1050 °C corresponds to an anomalous high diffusivity of boron ($\sim 10^{-9}$ m 2 /s). Interaction of boron atoms with moving dislocations that realize the plastic deformation at the micro crack tips is evidently the main reason of this effect which develops as boron “sweeping out” under tensile test.

Simultaneously in this area has been revealed the nucleation a set of micro cracks evidently at the weak grain boundaries after a small plastic strain.

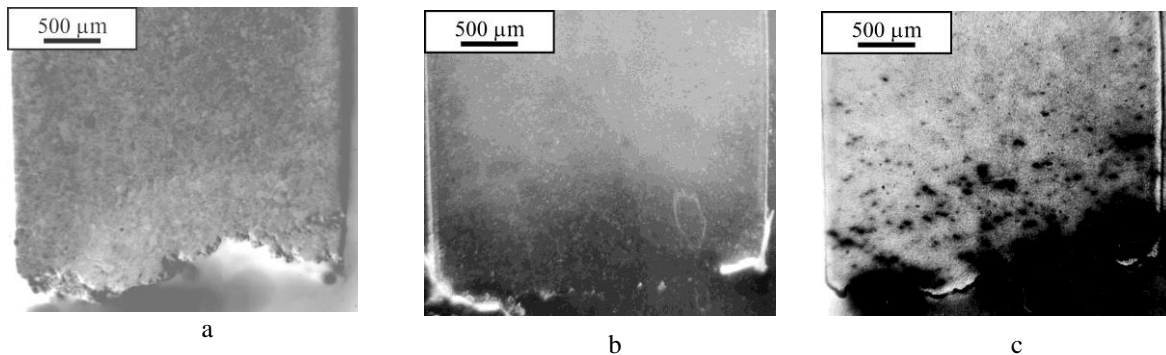


Figure 1. Macrostructure (a), track and activation autoradiographs of boron (b, negative image) and carbon (c) distribution in the fracture zone of the specimen of the rapidly quenched PM HIP Ni-based superalloy EP741NP after the tensile test at the temperature 1050 °C and strain rate 10^{-3} s^{-1} .

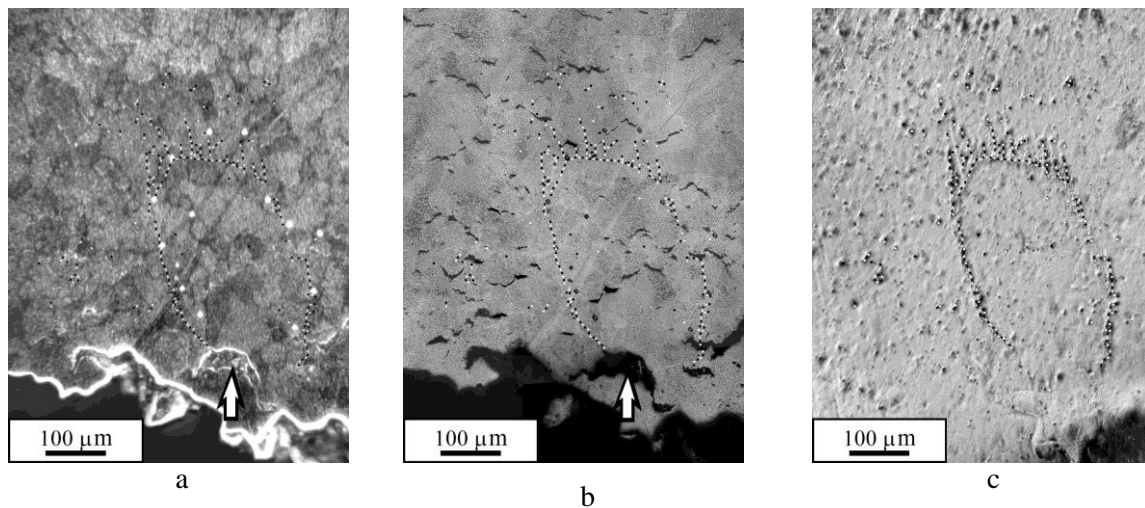


Figure 2. Microstructure (a, b), track autoradiograph of boron distribution (c, positive image) near the metho crack in the specimen of the PM HIP Ni-based superalloy EP741NP.

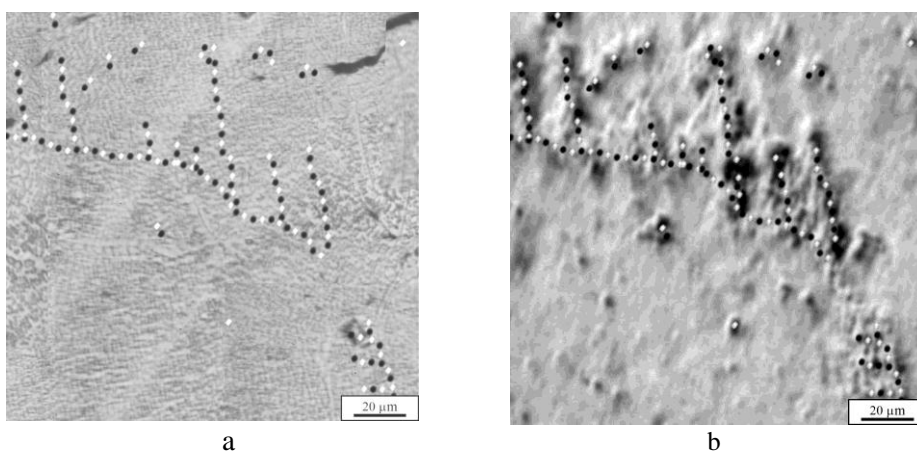


Figure 3. Submicrostructure (a), track autoradiograph of boron distribution (b, positive image) near the metho crack depicted by the black-white dot lines in the specimen of the PM HIP Ni-based superalloy EP741NP at the high magnification.

These micro cracks don't propagate to the fracture of specimen evidently because other grain boundaries are stronger and also owing to plastic deformation at their crack tips. A few larger intergranular (metho) cracks were nucleated during continued plastic deformation at the certain boundaries.

The specimen after the tensile test at the temperature 1050 °C and strain rate 10^{-3} s^{-1} is characterized by the metho crack (indicated by the arrow in Fig. 2) which provides the formation of the oval-shaped trail decorated by boron segregation. An examination of this metho crack, which did not lead to fracture, shows that the crack was initiated in an intergranular fashion. Whereas this crack was not propagated to fracture of the specimen, therefore this confirms that plastic deformation at the crack tip is the main reason of boron segregation. Intensive dislocations glide at the surface of maximum shear stresses near this crack tips and interaction of the moving dislocations with boron atoms evidently results in formation of the oval-shaped trail enriched with boron. Atmospheres of boron atoms on the moving dislocations form well known solute atmospheres produce a drag force to moving dislocations [12, 13]. Under considerable deformation, the crack size becomes large enough to cause fracture resulting in the mixed intergranular and ductile tearing fracture mode.

The multiscale study of boron distribution in the fracture zone of the specimen of the PM HIP Ni-based superalloy EP741NP after the tensile test was revealed decreasing the density of borides precipitates of the type (W, Mo)B according to the in the fracture zone simultaneously with depletion of the track density in this area. Decreasing of track density in the fracture zone on the track autoradiograph on boron distribution indicates the decreasing of boron atoms in solid solution. Whereas boride precipitates are characterized by the coincidence of separate tracks from boron atoms on the track autoradiograph with formation of fine spots which correspond to the fine boride particles. Therefore decreasing of this spots density on the track autoradiograph means the decreasing of the boride particles density under tensile test at 1050 °C.

After the tensile tests at the lower temperatures, for example at 800 °C, was detected by the track autoradiography, scanning electron microscopy, and x-ray spectral microanalysis the homogeneous boron distribution without depletion of boron and dissolution of dispersed boride particles of the type (W, Mo)B in the fracture zone of the specimen. Thus, the significantly lower solubility of boron at this temperature evidently is the main reason of this effect.

An activation autoradiography investigation of carbon distribution in the fracture zone of the tested specimens allows detecting the absence of significant change of carbon distribution as it was detected in the case of boron. The main reason of this difference is evidently the essentially higher thermal stability of carbide phases in the nickel-based superalloys when compared with the thermal stability of boride phases. The results of the study of boron and carbon distribution in the fracture zone of the rapidly quenched nickel-based superalloy by the nuclear physics methods of the track and activation autoradiography demonstrates essentially different effect of crack propagation on boron and carbon distribution.

A detailed study of carbide particles distribution by scanning electron microscopy and x-ray spectral microanalysis in addition to the activation autoradiography in the fracture zone was confirmed an essentially higher stability of type (Nb, W)C carbide phase then boride phase (W, Mo, Cr)B type particles distribution under the high temperature tensile tests. Therefore carbon and carbide particles maintain resistance to fracture under the high temperature tensile test in significantly higher degree when compared with boron and boride particles effect.

4. Conclusion

In this work behavior of boron and carbon in the specimens cut from the rapidly quenched PM HIP compact of the Ni-based superalloy EP741NP were investigated. Multiscale study (at macro, metho, micro level) of boron and carbon distribution by autoradiography methods on boron and carbon simultaneously with metallography, Scanning Electron Microscopy (SEM), and X-ray Spectral MicroAnalysis (XRSMA) (nano level) were performed. The significant effect of fracture process on the boron distribution in the fracture zone and the microstructure of the rapidly quenched PM HIP compact of the Ni-based superalloy were determined. It was established the followings:

1. Significant decreasing of boron concentration in the fracture zone of the specimen of the rapidly quenched PM HIP Ni-based superalloy compact after tensile test at 1050 °C and strain rate 10^{-3} s^{-1} was

detected. The length of boron depleting area reaches one millimeter in direction to the fracture surface. Simultaneously in this area has been revealed the nucleation a set of micro cracks evidently at weak grain boundaries after a small plastic strain.

2. Essential boron depletion in the fracture zone under testing at the temperature 1050 °C can be explained as an anomalous high diffusivity of boron. Interaction of boron atoms with the moving dislocations that realize the plastic deformation at the crack tips is evidently the main reason of this effect. Some influence of the dilatation strain in the near tip field occurs evidently on the boron migration.

3. Influence of the test temperature on the features of boron distribution in the fracture zone of the rapidly quenched PM HIP Ni-based superalloy EP741NP was also investigated. After tensile tests at the lower temperature, for example at 800 °C the homogeneous boron distribution without depletion of boron in failure zone was detected.

4. In the fracture zone was revealed an essentially different behavior of boron and carbon, which are both the microalloying interstitial element. Carbon distribution in fracture zone of tested specimens indicates essentially higher stability when compared with boron distribution.

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