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NEW TECHNIQUE FOR CHARACTERIZATION OF MICROSTRUCTURE OF THE NICKEL-BASE SUPERALLOY

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Abstract: *A new short-in-time and effective experimental technique (hard cyclic viscoplastic deformation of gradual shaped specimens) was proposed for characterization of microstructural evolution and accumulation of fatigue damage under cyclic loading condition. This technique was applied to investigate the dendrites microstructure change via deformation diffusion of the single-crystalline nickel-base superalloy ZS-32vi in a wide range of strain and stress levels at room temperature. The results show that accumulated fatigue damage predicts the reduction of Young's module and increase of plasticity.*

Key words: Nickel-base superalloy, single crystal, microstructure, cyclic deformation, diffusion.

1. INTRODUCTION

Well known that the microstructure and mechanical properties of engineering materials degrades during service time and thereby reduce the lifetime of structural components. This problem is conclusive for single-crystal (SC) turbine blades material of the turbo-jet gas turbines operating in gas surroundings and hard cyclic loading at high temperatures. The thermal and mechanical loadings of rotor blades during exploitation, generally involving centrifugal force and buffeting at gas surroundings are complicated [1]. According to this the mine areas of investigation of these materials mechanical properties behavior at elevated temperatures are: tensile creep deformation and rafts forming [2-4], thermal-mechanical fatigue (TMF) [5,6], multiaxial high cycle fatigue (MHCF) [6-8] and low-cycle fatigue (LCF) [9]. Such large amount of powerful test methods can thus improve the turbine blade manufacturing technologies, increase inlet temperature before turbine as well as life time of turbo-jet. The test results as well as blades manufacturing technique features have direct influence on SC superalloy development programmes. These programmes [10,11] are canalized to develop the directional solidification techniques at various thermal gradients and solidification rates as well as chemical condition optimize. For manufacture of SC superalloys are used directional solidification equipments with molten Ga-In liquid metal cooling and high costly refractory metals as alloying elements such as W, Ta, Re, Cr, Mo, Nb, Co, Yt, Hf, Ru, B, C, etc. Regardless of these presented above classical and high costly test methods these materials nevertheless up to here were not tested in viscoplastic conditions [12-14].

In this paper a new experimental technique is introduced which allowed to reduce significantly testing time and number of specimens for testing, as the gradual shaped specimens were proposed. Therefore, the aim of this study is to present new experimental technique for characterization of microstructural evolution and mechanical properties degrade as well as fracture mechanism at followed tension for application to the new fifth generation single-crystalline nickel-base superalloy ZS-32vi.

2. EXPERIMENTAL

The experimental material used in this study was SC castings of the Ni-base superalloy ZS32-vi. The nominal composition of the superalloy in atomic per cent (at%) is as follows: Al 12.1, Cr 5.3, Co 9.4, Mo 0.7, Nb 0.85, Re 0.9, Ta 0.9, W 0.24, and Ni balance [15]. Castings were produced by means of electro-induction melting method in a directional solidification furnace under a high vacuum and solidified in the (001) direction within a deviation $\leq 4^{\circ}$ off. The solidification rate was about 8.8 mm per minute as the dendrites in virgin casts were up to 5 mm in length [10]. The conventional SC creep testing samples (Fig. 1) with a gripe section diameter of 15 mm and the starter block (SB) at the conical end with overall length of 120 mm were manufactured. The test part for creep testing has diameter of 10 mm and length of 60 mm. The previous vacuum aging heat treatment at 1280 °C for 1.5 h was conducted. In the present study in the middle part of the sample was manufactured test part with diameter of 7 mm, with fillet radiuses of 2 mm and length of 12 mm for (tension-compression) testing (Fig. 1). The dotted line represents conventional creep properties testing sample geometry. The chemical condition testing zones 1, 2 and 3 are indicated. The step-by-step decreased cross-section areas: 1─176 mm^2, 2─78.6 mm^2 and 3─38.5 mm^2 are indicated in the sample, respectively. During straining the different cross-sections are loaded proportionally under different stresses and strain levels, respectively.

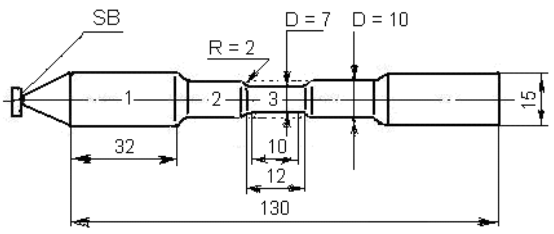

Fig. 1. Schematic illustration of the test specimen geometry.

The samples (with gradual cross-sections) testing were performed under the strain amplitude controlled regime on materials testing system Instron-8516. For a true strain measure, the extensometer with base length of 10 mm was used. The pre-fatigue testing (according to proposed new test method) was conducted under the tension-compression loading at room temperature. The three strains and adequate isothermal stress histories are studied for verification purposes as depicted in Fig. 2. For this, a wide range of strain rates was controlled how the strain rate has an effect on the microstructure evolution to great extend of the SC superalloy. The loading with steps of strain amplitude of 0─0.05%; 0─0.2%; 0─0.5% and 0─1% as well as at strain amplitudes of ±0.05; ±0.2; ±0.5 and ±1% for each strain value and for 30 cycles was conducted, respectively. For each cycle the 100 test points of displacement and load for cycle number and time were automatically measured. The tension-compression loadings were conducted under low frequency of 0.5 Hz and, consequently at increased strain rate for each step whiles the strain amplitude was increased.

Before and after cycling the test specimens were tested at tension up to 0.2% and the Young's module at strain of 0.1%, 0.2% and "automatic" was measured. For study of microstructure evolution and fracture mechanism of fatigued material the fourth condition was examined by tension up to fracture.

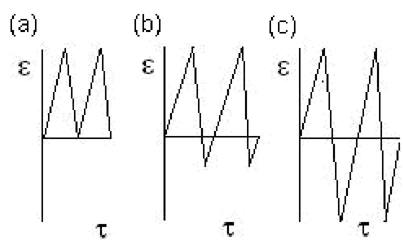

Fig. 2. Simple isothermal triangular time-strain histories of cycling: a- tension only, b- tension with compression after elongation at first cycle, and c- tension-compression symmetrical straining.

For the study of microstructure evolution during straining, the cuts, which were cat off from different part of sample (see Fig. 1) after straining were mechanically polished and etched. For dissolving out of γ'-precipitates the chemical etching (33 ml HNO_3 and 66 ml HCl) was applied. The dendrite macroarchitecture of samples was analyzed by using the light optical microscope (OM) Nikon Microphot-FX. The microstructure, phases chemical conditions (EDS) and fracture surfaces of samples were analyzed by the scanning electron microscopy (SEM) Zeiss EVOA15 and high resolution field emission (FE SEM) Zeiss ULTRA-55 equipped with the In-Lens secondary electrons (SE) detector for topographic imaging and energy, and the angle selective backscattered electrons (EsB) for the compositional contrast.

3. RESULTS AND ANALYSIS

The results of pre-straining at tension only of the SC superalloy are presented in Fig. 3. The pre-straining was conducted according to time-strain history (see Fig. 2, a) at strain amplitudes of 0─0.05, 0─0.2 and 0─0.5%, respectively. During pre-straining the sample of SC-1 show fully elastic behaviour at maximal stress values of 75, 300 and 760 MPa, respectively. The Young's module was not changed and was 150 GPa as constant for all test series (see Fig.3 and Fig. 5, curve EL). Unfortunately, at that time the low softening of tension stress at very first's cycles takes place. In addition the stress-strain curves for fourth test series (see Fig. 2, b) at strain amplitude of 0─1% for 30 cycles in Fig. 4 are presented. At tension part of first cycle the plastic elongation from 0.64 to 1.04% takes place. As is shown in the diagram, the strain-stress curves (2 – 30 cycles) for strain values of 0 – 1% cover each other. The tensile curves up to fracture (Fig. 5) and stress amplitudes (Fig. 6 and 7) are received according to presented new test method.

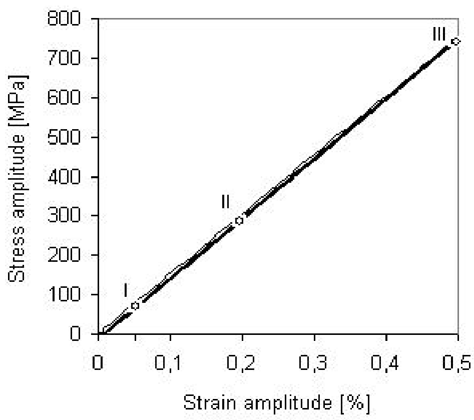

Fig. 3. Measured stresses obtained in each phase during elastic loading of the SC for triangular wavelength (Fig. 2, a).

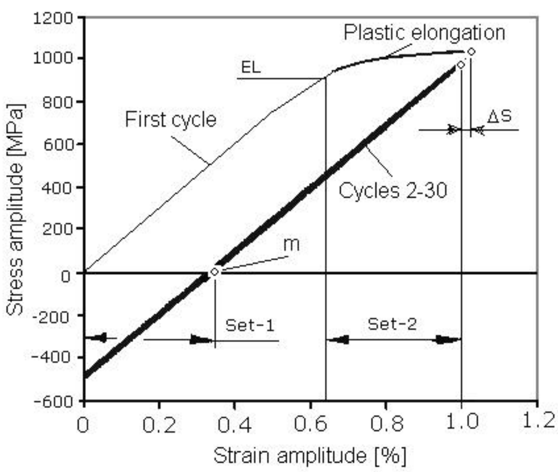

Fig. 4. Stress-strain curves for triangular wavelength with plastic elongation from 0.64 to 1.04% at first cycle.

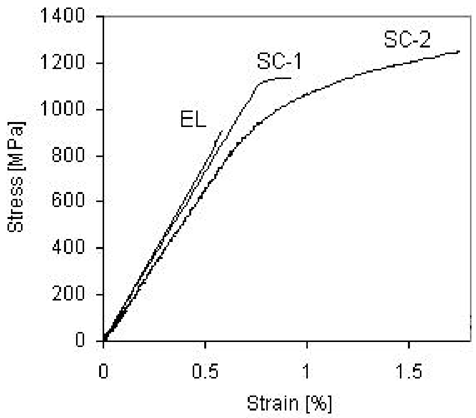

Fig. 5. Tensile curves up to elastic limit (EL) at first cycle and up to fracture (SC-1, SC-2).

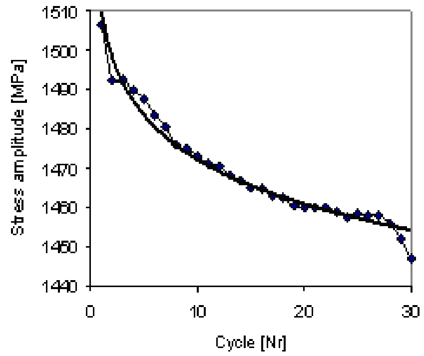

Fig. 6. Decrease of the stress amplitude during cycling at strain amplitude of 0─1% for 30 cycles (scheme b ─ Fig. 2,).

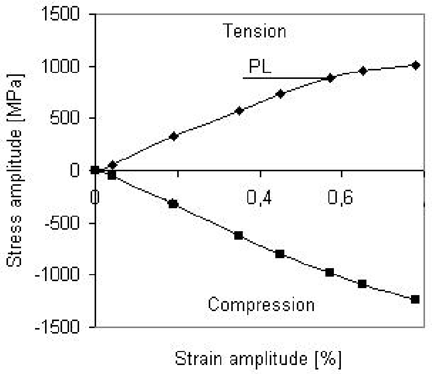

Fig. 7. The dependence of stress amplitude increase during HCV deformation (scheme c ─ Fig. 2) on strain amplitude absolute value increase.

The measured at tension Young's module values were step-by-step (150, 147, 143 and 134 GPa, respectively) decreased according to increase of collected strain.
The dendrite microstructure of virgin SC in Fig. 8 is shown. The microstructure (see Fig. 1, region 1) is characterized by large dendrites with length up to 5 mm and large regions of fragile γ-eutectics, metal carbides (MC) and Nb-Ta intermetallic between dendrite arms. As result of HCV deformation the dendrites measures were decreased (Fig. 9) from 5 mm to 0.3 mm in length (~15 times). The γ-eutectics measures were decreased too from ~100 to ~40 μm in mean. Such microstructure evolution aided by deformation diffusion under HCV deformation has been studied earlier in [13,14].

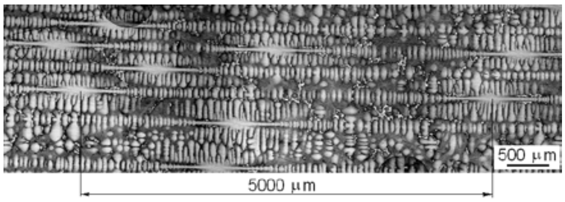

Fig. 8. The optical image of the dendritic microstructure of virgin SC Ni-base superalloy.

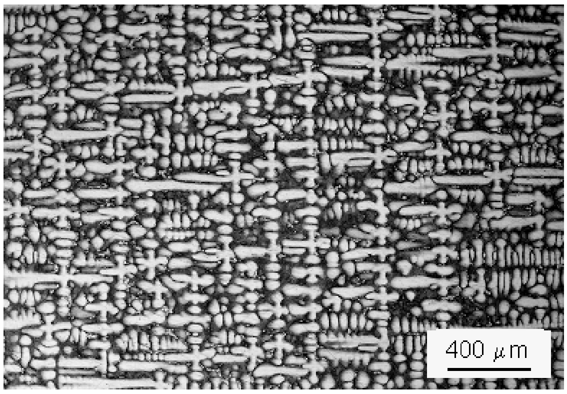

Fig. 9. The optical image of the dendritic microstructure of SC Ni-base superalloy after HCV deformation.

Well known [15,10] that reducing the dendrite arm spacing measures has direct influence in reducing the size of eutectic and to improve the mechanical properties of SC superalloy. For study this phenomenon of microstructure and mechanical properties change the material in these regions with different cross-sections were studied by FE SEM microscope. Early in [13,14] was shown, that chemical conditions change via deformation diffusion like thermal gradient and solidification rate [10] has influence on dendrite arm spacing and size of eutectic.
Under cyclic tension-compression loading at room temperature, the nickel base superalloys are subjected to microstructural evolution [13]. The most commonly known evolution is the rafting [2,4] of the precipitates along the <001> direction (Fig. 10). In this situation, the γ'-precipitates, initially cuboidal, coarsen in an anisotropic way to form platelets parallel to the tensile axis.

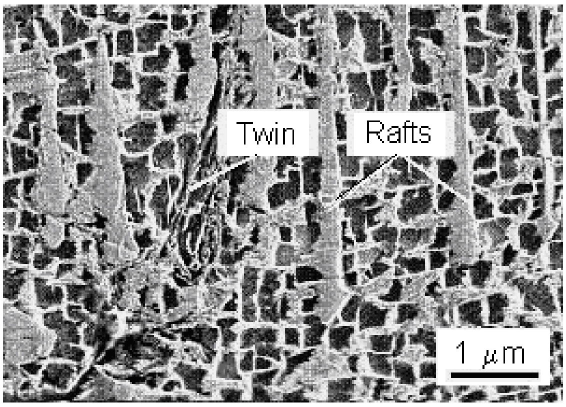

Fig. 10. Morphology of the HCV deformed SC with twin and rafts forming.

The development of rafted structure strongly influences the mobility of the dislocations and twins [4,6] and then the rate of strain accumulation especially at relatively low applied stresses, when cutting of the reinforcing particles is difficult.

The experimental results indicate that viscoplastic deformations and fracture mode depend on the evolution of the microstructure. The possible difference in chemical composition between the dendritic and interdendritic regions was investigated by EDS. The measurements reveal a bimodal chemical elements distribution (Fig. 11). The results show very good correlation with the dendritic and interdendritic regions [11].

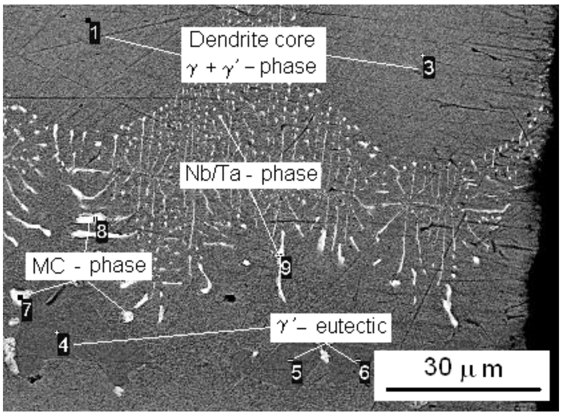

Fig. 11. Electron image of EDS investigation near fracture surface of SC after HCV deformation. The points of chemical elements measure are depicted.

The role of refractory alloying elements such as W, Nb, Ta and Re on basis of the active deformation mechanisms and of the respective strengths of the γ- phase matrix and the strengthening of γ'-precipitates is important. Unfortunately, these γ'-precipitates are fragile (with compare to γ-phase matrix) and fracture at first at subsequent tension. In SC Ni-base superalloys both diffusion and dislocation based creep contribute to inelastic deformation as well as to the evolution of the γ- and γ'-phases microstructure. It was found from the uni-axial HCV deformation experiments that the plastic strain at room temperature could change the microstructure in a same manner of the rafting phenomena. It was also found by the experiments using the gradual shaped specimen, in which residual stress remained, that the directional rafting was governed by the magnitude of residual stress as well as the sign rather than the plastic pre-strain itself. Importantly, improvements in the fundamental understanding of structure evolution during processing and its relationship to properties also follow to result in materials improvements.

4. CONCLUSIONS

This new low cost and effective destructive test method (conducted at room temperature) has large tension-compression step-by-step increased strain amplitude, low frequency and small reduced number of cycles.

HCV deformation is a novel technique to improve of SC Ni-base superalloys plasticity via dendrite spacing decrease. The withdrawal speed and furnace temperature [10,11,15] have a predominant influence on the dendrite arm spacing as the HCV deformation at room temperature has.

The deformation diffusion has great influence on the microliquation especially for the very fine dendrites length and arm spacing.

Rupture life and plasticity of fine structured samples (under HCV deformation) increase significantly than that in virgin sample, indicating that it is an effective way to improve mechanical properties of SC superalloys.

The HCV deformation is beneficial method to refine the dendrite arm length from 5 mm to 350 μm in mean. Increased strain amplitude was also effective to reduce the dendrites length.

As expected, the mirosegregation of elements Ta, W and Re and size of dendrites are significantly suppressed.

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