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Report:

"In Situ" Determination of γ' Phase Volume Fraction and of Relations between Lattice Parameters and Precipitates Morphology in Ni-Based Single Crystal Superalloy

submitted to Acta Materialia

abstract:

Diffraction profiles of single crystal Ni based superalloy samples with different microstructures were measured "in situ" up to the complete solutionizing of the γ' phase, using high resolution triple crystal diffractometer and high energy synchrotron radiation (150 keV, λ=0.08Å). A comparison between non deformed sample and creep deformed specimens with various resultant microstructure evidenced a relation between the lattice parameter distribution, the γ' precipitate microstructure and the sign of the connectivity. It was shown that a deformation induces a change in the relative volume cell of γ and γ' phases. Moreover, the high resolution of the experimental set-up allows in many cases to measure with a good accuracy the γ' phase volume fraction.

Conclusion :

The high resolution of theTCD of the ESRF high energy beam line ID15A allowed accurate analysis of the AM1 single crystal superalloy microstructure from room temperature up to the complete solutionizing of the γ' phase. Two quantities were studied for reference and crept deformed specimens : the lattice parameter distribution and the γ' phase volume fraction. Several remarks can be drawn from these measurements:

1- the temperature behavior of the lattice parameter mismatch depends strongly on the deformation level. When rafts start to develop, the isotropy of the three (h00) directions is lost and a dislocation network take place in the raft plane to reduce the coherency stresses at the γ/γ' interfaces. At this first stage the lattice parameter mismatch is locked in the raft plane while in the perpendicular direction, lattice parameters can change almost independently in order to compensate the difference between the thermal expansion coefficients of γ and γ' phases. The absolute values of lattice parameter mismatches in both directions tend to increase with the deformation. This is pursued up to a deformation level for which a threshold in term of dislocation density in the raft plane, is reached. At this level if the rupture did not occur, dislocations start to settle also in the plane perpendicular to the raft and lock the lattice parameter mismatch also in this direction. This phenomena is accompanied by a strong distortion of the raft geometry. This behavior is enhanced when the deformation stress is weak and the temperature of the deformation is not too high : in this case the evolution of the microstructure is slow enough to let time to dislocation networks to settle on.

2- the difference in average unit cell volume of the γ and γ' phases was shown to be influenced by the deformation level of the specimen and the relaxation status of the γ/γ' interfaces. This parameter tends to increase with the deformation but for each stage level it seems to keep the same value.

3-the γ' phase volume fraction behavior in temperature was determined with a high accuracy from the analysis of crept deformed sample. It was shown that this parameter does not depend directly on the thermo-mechanical history of the material. The solutionizing of the γ' phase starts in main cases around T = 950°C and its rate increases near T = 1150°C. It is complete above T = 1275°C. Comparison with previous neutron experiments and with preliminary synchrotron measurements of superstructure reflections shows that the order of the γ' phase depends significantly on the temperature and on the thermomechanical history of the material.