EXPLORATION OF LABORATORY X-RAY TOMOGRAPHY FOR IN-SITU RESEARCH OF VOID EVOLVEMENT

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Summary: Laboratory x-ray tomography (LB-XRT) using tube source is limited its availability for in-situ research due to poor brightness and monochromaticity. However, it can still cover abroad range of in-situ investigation of relative stable structure with slower or repetitive external change. In the cases of the voids in the alloys, it clearly revealed their evolvement in the thermal and electrical fields.

1. INTRODUCTION

Both laboratory and synchrotron radiation x-ray tomography (SR-XRT) are viable for in-situ experiments. The x-ray brightness from tube source is about ten orders magnitude less than that from synchrotron radiation. The setup for an in-situ experiment using LB-XRT has to be considered beforehand. Micrometer sized voids as defects pre-existing in alloys during processing might affect mechanical performance in service. For the in-situ study of these micro-voids, the samples in millimeter size can be much more easily prepared and manipulated than those in hundreds or tens of micrometers. In consequence, the energy needs to reach one hundred keV with polychromatic X-ray tube source, especially for alloys contain heavy elements. The imaging resolution and the field of view have also to be considered for the study of the voids morphology evolvement. The temporal resolution of LB-XRT must be in the same time scale so that the structure can keep stable when the specified condition is applied [1].

In one case of a Ni-based super alloy [2], micro-void might be the key factor that influence the lifetime during fatigue and creep test. Micro-voids pre-existed during solidification and changed shape with solution treatment later on. Their evolvement during the thermal treatment was a typical slower process that is feasible object for in-situ LB-XRT study. Different behavior of morphology change for two types of the micro-voids was demonstrated. In the other case of a high-strength steel [3], the micro-voids might make trouble for continuous plastic flow with strain-hardening and induce failure during deformation. Nevertheless, the micro-voids might be healed by electropulsing treatment (EPT). The matter migration driven by repetitive electrical stress field was deduced from micro-void shape change that was revealed by in-situ LB-XRT.

2. EXPERIMENTAL METHOD

The Xradia Versa XRM-500 system in our laboratory was employed for the in-situ study of the micro-void. The voltage of the x-ray tube with micro point source was operated up to 150 keV. The x-ray with a cone beam transmitted through the sample and the magnified projections was then converted to visible light by a scintillator and imaged by a 2 k × 2 k CCD camera. 960 projections with a 360° rotation were taken for one 3D volume with a pixel size about 1 μm and volume size about 1 mm^3 for the investigation of the micro-void in the Ni-based super alloy and the steel for EPT treatment. All of the datasets were performed a correction of beam hardening and then reconstructed by the bundled software kit with the filtered back projection (FBP) algorithm. Reconstructed 3D tomography data were visualized and processed with Avizo software, through which segmentation and quantitative analysis can provide clear and solid information of the micro-voids. The samples were carried on LB-XRT back and forth after the repeated thermal or EPT treatment in an interrupted mode for the in-situ research. Special set up to fix the same sample position for the LB-XRT was used to correlate the same views of the interested micro-voids.

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3. RESULTS

The LB-XRT uncovered two types of micro-voids and can separated them from their shapes and the morphology evolution during thermal treatment. One type was pre-existing voids after solidification and their volume fraction changed with solution thermal treatment at 1603K. This type of micro-void decreased the volume fraction in about 4 hours, and then increased the volume fraction with extension of treatment time from 7 to 20 hours. The other type of micro-voids formed and grew during the solution treatment and with extended treatment time. It was indicated that the cross-diffusion of elements in multi-component alloys might induce vacancy formation and accumulation, which originated the generation of the micro-voids.

The LB-XRT demonstrated the morphology change of a long 3D crack in the steel specimen before and after EPT treatment. The overall length of the crack decreased from about 400 μm to 300 μm before and after EPT. Size shrinking for other micro-voids can also be observed. It was proved that macro voids in the interior of the steel can be healed by EPT. The healing effect of EPT may originate from two aspects: one is the elevated temperature near the crack, and the other is the thermal compressive stress around the crack. The research of the crack healing by in-situ LB-XRT could be expanded to investigate other damage recovery for engineering alloys.

References

[1] L. Zhang & S.G. Wang, Correlation of Materials Property and Performance with Internal Structures Evolvement Revealed by Laboratory X-ray Tomography, *Materials*, 11, 1795, 2018.

[2] X.W. Li, L. Wang, J.S. Dong, L.H. Lou & J. Zhang, Evolution of micro-pores in a single-crystal nickel-based superalloy during solution heat treatment. *Metallurgical and Materials Transactions a-Physical Metallurgy and Materials Science*, 48A, 2682-2686, 2017.

[3] C.L. Yang, H.J. Yang, Z.J. Zhang & Z.F. Zhang, Recovery of tensile properties of twinning-induced plasticity steel via electropulsing induced void healing, *Scripta Materialia*, 147, 88-92,2018.

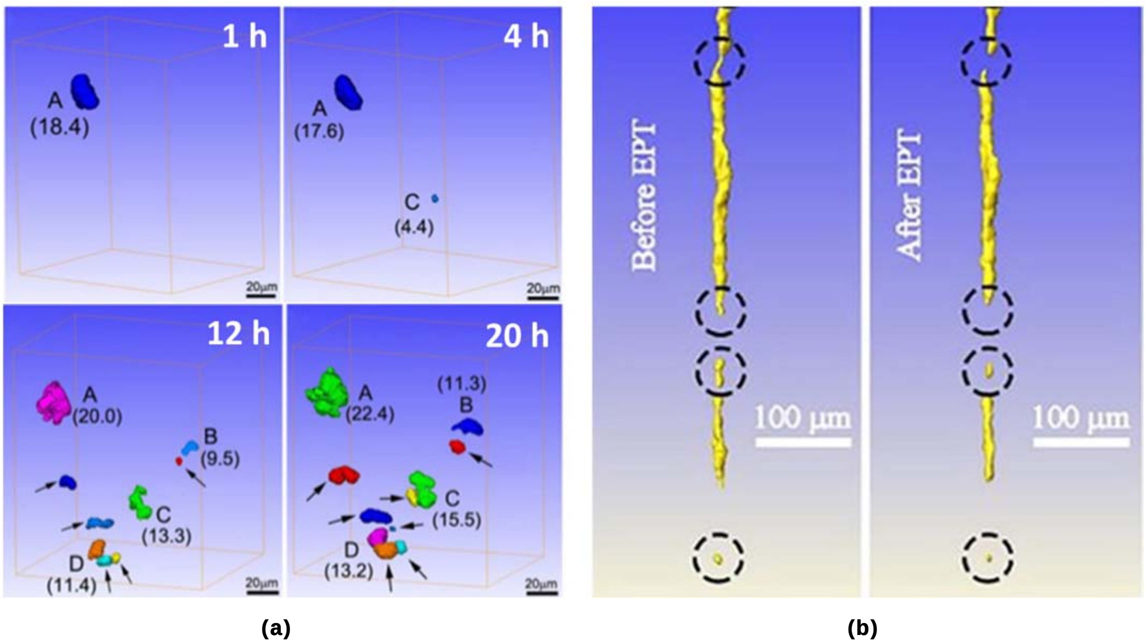

Figure 1 In-situ LB-XRT results of micro-void evolution (a) in the Ni-based super alloy during solution thermal treatment and (b) in the high strength steel undergoing electropulsing treatment.