Phase Contrast Imaging of Superalloys

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1 Ni-Based Superalloys

Ni based single crystal superalloys are used in fabricating gas turbine blades for the industrial and aerospace sectors because of their strength and resilience to extreme mechanical and temperature operation conditions. Aircraft turbine blades need to be able to withstand temperatures as high as $1000^{o}C$ under large mechanical stresses [1]. Alloys have been developed recently which can withstand temperatures as high as $1200^{o}C$. The growth conditions strongly influence the microstructural composition of the alloys which determine the overall performance the alloys.

Ni-based superalloys may be used either in the single crystalline form where the blades are cast from mold as single crystals or in the equiaxied phase which have a lower degree of crystallinity. Equiaxied alloys are usually used in the stages of turbine engines which are relatively cooler mainly because of the high cost of producing single crystal alloys [1].

A good understanding of the causes and development of failure in Ni-based superalloys is critical to developing a monitoring system to ensure that they do not fail under operation conditions. Fatigue and fatigue crack propagation are the usual candidates for damage. Physical models have been developed to predict defects in superalloys, however, these models have to go hand in hand with sensory systems to assess the 'damage state' [1] [2] of the material as efficiently as possible.

2 Phase Contrast Imaging

To image and study the the microstructure and the causes and types of defects which lead to the failure of Ni superalloys, a number of techniques such as Scanning Electron Microscopy, Transmission Electron Microscopy and X-ray Fluorescence spectroscopy have been employed. These methods have proven valuable in imaging Ni superalloys up to the nanometer scale. However, x-ray imaging is the only technique which allows for non-invasive three dimensional observation of internal features. [3]

2.1 Benefits of Phase Contrast

Traditional x-ray images are produced in which the contrast of the image is strongly dependent on the variation in absorption of x-rays by the different constituent elements of the material being imaged. Absorption contrast, however, does not provide enough sensitivity to distinguish phases in a material which have similar x-ray attenuation properties or the imaging of low x-ray absorbing materials Microsegregation occurs in most alloy systems and provides additional contrast which is generally not revealed by conventional x-ray absorption imaging.

Phase contrast imaging in recent years has been shown to be a superior method for increasing the contrast in x-ray tomography and radiography [4][5][6]. Contrasts are produced by interference between parts of the transmitted wavefront that have undergone different phase shifts, due to local density inhomogeneities. Phase contrast imaging has enabled imaging of low density materials and materials which contain different elements with similar electron densities. Combined with diffraction, phase contrast imaging allows for applications such as the study of compressive stresses, in-situ dendritic growth [6] [7] and the tomographic investigation of defects in materials.

Phase Contrast imaging techniques provide an excellent non-destructive way of directly probing/imaging Ni single crystal superalloys by utilizing the high spatial and temporal coherence and brilliance available at x-ray synchrotron sources. Recent studies have shown that it is possible to image Ni-based superalloy samples with thicknesses up to $300\mu m$ with resolutions on the micron scale and contrast high enough to clearly observe directly grain boundaries and compositional variations within the dendrites which are characteristic of directionally solidified Ni-based superalloys.

2.2 Requirements for Phase Contrast Imaging

Phase contrast imaging requires that the beam used for imaging has good spatial coherent, since the technique is strongly dependent on the coherent diffraction and interference of the transmitted beam.

Third generation synchrotron sources are an excellent source of coherent x-rays because of the monochromaticity of the x-rays produced and the small source size ($> 100 microns$). Although the resolution is in the micrometer range, the low divergence ($0.1 - 1\mu rad$) allows for imaging large volumes (in the mm^3 range) at large sample-detector ranges with a large sensitivity ($< 10^{-8} - 10^{-3}$). The energies available at synchrotron sources are tunable from the infra-red range to the MeV regime. The brilliance (up to 10^{12}) of synchrotron sources makes imaging acquisition possible in fractions of seconds making time resolved studies possible.[8]

The high resolution provided in synchrotron imaging experiments would not be possible if high resolution detectors were not available. CCD cameras with high readout rates, low noise and fast readouts are commonly used in synchrotron imaging. High resolution films are also used in some experiments.

2.3 Phase Contrast Microtomography

Microtomography experiments involve taking a series of images of a sample while rotating the sample through 180 degrees. The images can then be reconstructed to obtain a three dimension image of the object.

A few challenges arise when coherent beams are used[9]. A relatively straight forward procedure can be used in reconstructing images obtained using incoherent beams for imaging. However in phase contrast additional phase objects have to be accounted for in reconstructing images when coherent beams are used. Theoretical and experimental work has been carried out by Piex et al to account for this effect [3]. The result is a 3D image in which features are revealed which are not accessible in absorption contrast tomography.

Care has to be taken to avoid disturbing the wavefront of coherent x-rays before they reach the sample. Many beamlines include beryllium windows which introduce artifacts in the final images which are difficult to deconvolute. This type of artefact can be removed by using random phase screens, but at the cost of reducing the beam coherence [10].Also, in imaging crystalline materials, Bragg diffraction is unavoidable and care has to be taken in reconstructing the images.

2.4 Methods of Reconstructing Phase Images

Phase contrast imaging shares the property of holography, in that, three dimensional information is encoded in a 2 dimensional image. The phase function is dependent on the energy of the x-rays, the sample-detector spacing and most importantly, the shape and dimenension of the phase object [4] [5]. By taking images with different sample-camera separations, it is possible to obtain 3D information about the sample being imaged. A considerable effort has been undertaken in developing algorithms to reconstruct objects from phase images.

3 Other Synchrotron work

The strength of Ni-based superalloys are strongly dependent on the volume fractions of the γ and γ' precipitates[2][1]. Since the microstructure plays an important role in the performance of superalloys, it is important to understand the growth process on a micron scale in-situ if possible. A lot of theoretical work has been carried out to model and understand the growth of dendrites. The unique properties of synchrotron radiation have been utilized to study in real time the dendritic growth of metallic superalloys[7]. Real time in-situ studies have been carried out on the dendritic growth of metallic alloys at the ESRF in Genoble, France. The experiments by Mattieson et al[7] have obtained images with 3 ms temporal resolution and 5micron spatial resolution. Using phase contrast imaging, the alloy solidification process has been studied.

X-ray Bragg diffraction is an important tool because it reveals information about residual and induced strain fields within crystalline alloys. Bragg diffraction has been used simultaneously with phase contrast imaging to directly study defects in metallic alloys. [11] [2]

4 Conclusion

The development of an efficient complete set of tools to understand and predict failure in Ni-based superalloys is crucial. X-ray imaging and diffraction techniques provide a direct non-destructive way of probing superalloys. The availability of coherent and brilliant x-ray sources have opened the door for boosting the sensitivity of x-ray techniques through phase contrast. Images and tomographs can be obtained in a mater of seconds with sub-micron spatial accuracy. The additional sensitivity to strain fields makes imaging fatigue and fatigue cracks possible.

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