論 文 の 内 容 の 要 旨

論文題目Development of High Temperature Local Strain Measurement System and Application to Al_2O_3

高温下での局所ひずみ計測技術の開発とAl_2O_3への応用

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Polycrystalline Al_2O_3 is most widely used as high temperature engineering ceramics. Fracture behavior of Al_2O_3 is affected by intrinsic microstructural parameters such as grain size, defect size and degree of orientation as well as extrinsic factors like loading condition, atmosphere and service temperature. Specifically, α-Al_2O_3 has significant anisotropy in coefficient of thermal expansion (CTE), causing thermal stress between grains. Inhomogeneity of structure and stress distribution in the material on the order of grain size, typically micrometers to several tens of micrometers order, play important roles in crack nucleation and crack extension behavior before unstable crack propagation. Thus in-situ observation of time-dependent and temperature-dependent deformation/fracture behaviour of polycrystalline Al_2O_3 and full-field strain measurement are of significant importance for safe usage of Al_2O_3 materials for structural use at high temperature. The purpose of this thesis is to develop an in-situ observation and local strain measurement technique available up to 1400 °C using optical microscopy with UV illumination. The required performance for the technique and design concept of the new system were discussed. The system was constructed based on the design and the potential of the established system was evaluated.

In Chapter I, the significance of in-situ observation and strain measurement of Al_2O_3 on the order of micrometers at high temperature was proposed. Currently used techniques for in-situ observation and full field strain mapping at high temperature were summarized and their advantages and disadvantages were compared. Through the consideration of the performance of current techniques and the required properties of (1) high temperature observation up to 1400 °C, (2) micrometers order resolution, (3) observation field ranging from millimeters to hundreds of micrometers, (4) strain accuracy of 10^{-5} order, (5) long term observation and (6) changeable atmospheres, application of optical microscopy and digital image correlation (DIC) were selected. Problems to be solved in the development of new system were then considered.

Summarizing the discussion, the purpose of the study was clarified and the content of the thesis was illustrated.

In Chapter II, according to the requirements presented in Chapter I, the concept of the new optical system was proposed. Based on the Planck's radiation law, the black body radiation intensity at UV light region above 1200 °C was discussed and the possibilities of using UV light as a light source in optical microscopy was considered. According to Planck's radiation law, intensity of thermal radiation is increased and the peak wavelength is shifted to a shorter (i.e. from infrared to visible) region with temperature increasing, which makes surface observation by conventional optical microscopy difficult. The radiation of UV light from a heated sample was found to be very weak up to 1400 °C, however. Thus it was proposed that an UV light was used as illumination in optical microscopy while the radiation from the heated sample ranging from visible to infrared region was removed by a filter. UV illumination can also provide a higher spatial resolution. According to the design concept, the required components in the new optical system were determined: an UV CCD camera, UV light source, optical microscope and UV bandpass filter. After selecting the components, the new optical system was constructed (designated as UV-HTOS). It has observation area ranging from 4.36 mm × 4.37 mm ~ 769 μm × 767 μm, resolution of 0.8 μm/pixel, maximum image size of 1004 pixels × 1002 pixels, satisfying the required properties. An indentation on polycrystalline Al_2O_3 plate was observed clearly without the influence of the thermal radiation at 1400 °C, showing the effectiveness of the proposed design concept and UV-HTOS constructed by the concept for the high temperature observation. Heating of the objective lens by a furnace was reduced by blowing air between the lens and observation window of the furnace. Long term observation is supposed to be possible at high temperature.

In Chapter III, the detailed evaluation of UV-HTOS was done by in-situ observation of the indentation on the same material of polycrystalline Al_2O_3. Heating –cooling cycles were conducted on the indented sample. The first cycle was in-situ observed using the conventional optical system without filter using the visible and infrared light. Before and after the heat cycle, the sample was observed by scanning electron microscopy (SEM). The healing behaviour of the indentation crack observed during the heat cycle was analysed and compared with the observation result by SEM. The conventional system captured a high contrast image and could observe the indentation crack to its tip at room temperature. The contrast decreased with increasing temperature and the image was oversaturated above 1100 °C due to the intensified thermal radiation, however. Then the second cycle was in-situ observed using UV-HTOS. The contrast of the image obtained by UT-HTOS was kept up to 1400 °C. Crack opening displacement of 1.2 μm at the crack root was accurately measured at both room temperature and 1400 °C. Crack length was measured accurately at room temperature but it appeared shorter than the actual length at 1400 °C. It is probably because the tip of the crack could not be observed due to the atmospheric turbulence at high temperature. Reduction of atmospheric turbulence was suggested to lead to further improvement of the system. Summarizing the result in Chapter II and III, it has been confirmed that UV-HTOS has a performance satisfying the requirement set at Chapter I, showing the potential for application to micrometer order DIC at 1400 °C.

For the measurement of strain by DIC, random speckle pattern on the surface is needed. However, methods for fabricating microscopic pattern stable above 1000 °C have not been well established. In Chapter IV, variety of micro-scale speckle patterns suitable for UV-HTOS observation up to 1400 °C were fabricated on the surface of Al_2O_3 plates: (1) NiO powder dispersed in either a solution of positively charged poly(diallyldimethylammonium chloride) (PDDA), negatively charged poly(sodium4-styrenesulfonate) (PSS), or deionized water was deposited on the Al_2O_3 plates by a spin coater; (2) commercially available Al_2O_3 or SiC paint was sprayed by air brush with different nozzle sizes; (3) the polished surface was abraded using 3 μm diamond slurry grinding medium on a lapping machine. The fabricated patterns were evaluated by mean intensity gradient (MIG), number of speckles, average speckle size and standard deviation of speckle size from the speckle size distribution histogram. The pattern fabricated by NiO powder dispersed in deionized water via spin-coater showed the largest MIG value of 45, the largest number of speckles, smallest average speckle size and smallest standard deviation of speckle size. Pattern fabricated by Al_2O_3 paint sprayed with an air-brush with fine nozzle size and the abraded pattern, showed fine and high density patterns with high MIG values of ~40. A simulation experiment was done by translating the image by image processing software and the translation was measured by correlating the original image and the translated image using DIC. The measurement errors by most of the fabricated patterns were less than 0.05 pixels, indicating that the expected stain accuracy was on the order of 10^{-5} when the observed area was 502 pixels x 501 pixels. It also shows that MIG alone is not enough to evaluate the pattern properties; a large MIG, even speckle size distribution and wide speckle size range pattern are required for a good DIC. Thermal stability of these patterns was evaluated by heating the patterns up to 1400 °C. The reaction between the patterning material and substrate resulted in the decrease of number of speckle patterns.

Cracking and blurring of the large paint pattern during the heating also change the surface pattern and may affect the DIC accuracy. Utilization of fine nozzle air brush in spraying the high temperature paint and abrasion of the polished surface resulted in the formation of small pattern stable at 1400 °C. They kept high MIG values and even size distribution after heated. The promising patterns for microscale DIC at high temperature has been proposed.

In Chapter V, the CTE of sapphire and thermal strain distribution of polycrystalline Al_2O_3 were measured by UV-HTOS using the high temperature random patterns established. Various furnaces at different heating/cooling rate were used for the purpose of comparison. The CTE of sapphire measured in an image furnace using the paint pattern was consistent with the literature data from room temperature up to 800 °C, showing the potential of the developed technique. Above 800 °C, decrease of image contrast due to the low emissivity of sapphire and increasing radiation from the speckle pattern; the heat haze between the furnace and objective lens; intensified radiation from the furnace lamp led to the deviation from the literature data. Determination of the pattern material considering the emissivity at high temperature and selection of the furnace should be done. Abrasion pattern on the polycrystalline Al_2O_3 heated in a Pt-wire heater furnace was clearly observed up to 1400 °C. Strain mapping was successfully obtained at 1400 °C over the entire region without analysis failure, though further improvement should be done for high accuracy.

The outcomes of the present study were summarized in chapter VI. Effectiveness of the achieved techniques for the understanding of thermal and residual starin distribution, microcracking and overall fracture of Al_2O_3 was described. The potential application fields of UV-HTOS utilizing high temperature in-situ observation and strain measurement were also mentioned.