Journal of Multidisciplinary Engineering Science and Technology (JMEST)
ISSN: 2458-9403
Vol. 3 Issue 11, November - 2016

The Study of SEM Examination of Crept Ceramic Samples Prepared by Cross Polishing Method

Alper Uludag
Faculty of Aeronautics and Astronautics
Anadolu University
Eskisehir, Turkey
alperuludag@anadolu.edu.tr

***Abstract*— Creep performance of a material is one of the major factors for determining its high temperature application. The determination of creep performance of a material is generally accomplished by experimental studies. In experimental creep studies, creep testing and microstructural analysis are used to determine the active creep mechanisms of materials being investigated. There are many cases where microstructural observation of a cross section is important and necessary. Scanning electron microscopy observation of flexure creep tested specimens is one of those cases.**

Cross section polishing method is one of the methods used for cross section sample preparation. In this study, microstructure of crept ceramics prepared by cross section polishing method was investigated by using scanning electron microscope.

Keywords— SiAlON ceramics; scanning electron microscopy; cross section polishing; Sample Preparation;

I. INTRODUCTION

The properties and behaviors of materials are generally determined by their microstructure. Therefore, microstructural characterization of the effects such as composition, processing, service conditions on materials microstructure has an important role in research and development and failure analysis studies. [1]. In the most of experimental studies related to materials science, different kind of mechanical testing and microstructural analysis methods have been implementing depending on their purposes. Some of those experimental studies are about high temperature material properties such as slow crack, oxidation and creep studies.

Creep is the time-dependent and permanent plastic deformation of a material subjected to stress at elevated temperatures and creep performance of a material is one of the major factors for determining its high temperature application. The determination of creep performance of a material is generally accomplished by experimental studies. In experimental creep studies, creep testing and microstructural analysis are used to determine the active creep mechanisms of materials being investigated [2].

Creep tests are normally performed by applying a constant load/stress and measuring the strain as a function of time at elevated temperatures and in different atmospheres such as air, argon, nitrogen etc. Creep tests can be conducted under various loading configurations such as tension, compression and bending. While metals are generally tested in tension, ceramic materials are tested in compression or bending due to ease in aligning and fixturing. After creep testing, microstructural analysis methods are applied to non-crept and crept materials to characterize creep behavior and active creep mechanism(s) and/or failure mechanism. Creep mechanisms of a material depends on its microstructure, stress and temperature [2-5].

Scanning electron microscopy (SEM) or transmission electron microscopy (TEM) has been widely used in the studies related to microstructural observations of materials. During microstructural analysis process, the structure of specimens is basically examined under different magnifications by SEM or TEM. The information content of the SEM and TEM is different. While SEM provides information relating to topographical features, morphology, phase distribution, compositional differences, crystal structure, and crystal orientation, TEM provides information relating to internal structure of the materials [6].

In the previous study, microstructure of SiAlON ceramics before and after creep tests were investigated [7]. In that work, creep tests were conducted on four-point bending method by using of an Instron 5581 creep testing machine. And then SEM analysis were performed on the cross section of crept samples prepared by mechanical polishing method. Different cross section sample preparation methods such as mechanical polishing, microtome, Focused Ion Beam (FIB), and Cross Section Polishing (CP) can be used for microstructural analysis [8]. In this study, microstructure of crept ceramics prepared by cross

section polishing method was investigated by using SEM.

II. MICROSTRUCTURAL CHARACTERIZATION BY SEM

Basically, a Scanning Electron Microscope (SEM) uses an electron beam to make an image. The electrons in the beam interact with the sample and various signals are produced. These signals can be used to gather information about the sample. Surface topography and composition are useful information. As the electrons interact with the sample, they produce electrons which are secondary electrons, backscattered electrons and characteristic X-rays (Fig. 1). These electrons are collected by one or more detectors. The detectors use these electrons to form images, and then images are displayed on the computer screen [9]. Fig. 2 shows scanning electron micrograph of silicon nitride (Si_3N_4) microstructure.

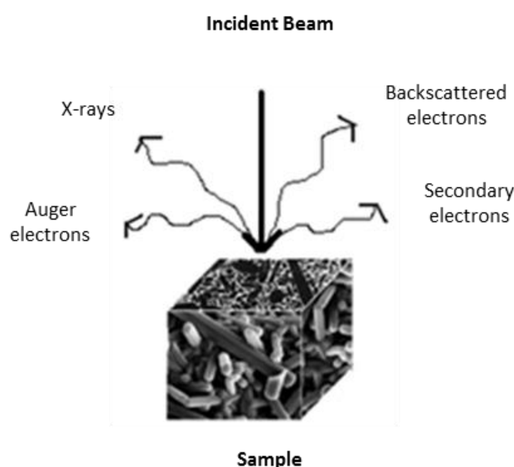

Fig. 1. *The electrons interactions with the sample*

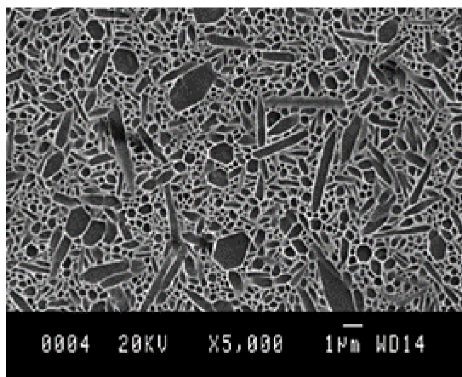

Fig. 2. *Scanning electron micrograph of Si_3N_4 [10]*

The use of SEM is the one of the most common methods for characterizing grain boundaries, fractures, cracks, flaws and defects on the surface of material investigated. Since SEM uses electrons coming from material's surface to form images, selecting sample and its surface preparation is very important for analysis. Actually, specimen preparation is the one of important stage in any type of microscopical method for the success of any subsequent stages. The examination and interpretation of microstructure of material can be done successfully, if the sample is prepared properly. Improper preparation of sample may lead to misinterpretation [11].

A. *General Steps of Sample Preparation for SEM Analysis*

There is no universal SEM sample preparation technique for all materials. Because each material has own nature, the sample preparation may be simple or elaborate. But, there are some general stages to prepare advanced ceramics for SEM microstructural analysis. These stages are selection of sectioning, mounting, grinding and polishing. Each of these steps must be optimized for each type of sample depending on hardness or nature of ceramic materials.

The first step in sample preparation is selection of sample for sectioning. The selected part should be representative of the material or deformed part to be investigated. The selected part are usually cut or sectioned to the area of interest by use of diamond cutting wheel or diamond wafer with a coolant. Some damage may occur while sectioning or cutting of sample and that damage can effect microstructure of material leading to misinterpretation. Therefore, proper selection and use of diamond cutting wheel or diamond as well as cutting speed, and load are important parameters influencing process.

Sectioned sample is then ground to expose a fresh surface. But it is usually necessary to do mounting of sample for handling easily and maintaining materials surface features before grinding. The mounting is done by encapsulating the sample into a mounting compound or resins. After mounting, sample is ready for grinding in a stable manner. Grinding is necessary to get a fresh and flat surface. In general, rotating disks of abrasive paper with coolant are used for grinding. After grinding, mounted sample is polished by using finer grades of diamond paste. The damage, which are occurred at cutting and grinding, can be reduced by polishing. Last stage is coating of samples by electrical conductive layer such as carbon or gold to prevent charge build-up on samples for SEM investigations. Because most of the ceramics are electrical insulators. In this study, selection of sectioning, grinding steps and cross polishing method are investigated in more detail.

B. *Sample Preparation of Flexure Creep Tested Specimens for SEM Analysis*

As it is stated before, the first step in sample preparation is to select a sample that is representative of the material to be evaluated. After selection, it was prepared for SEM analysis. The region or area of interest depends on test method such as tensile, compression or bending test.

The four point bending or flexure creep testing method has several steps. At the beginning of test, a specimen is supported on two supports near its ends. After it is heated to the required temperature, constant bending load or stress is applied to specimen by two loading points. The deformation or deflection of the test specimen is measured and recorded with time.

The loading configuration, deformed shape, stress and strain distribution occurring in bending testing are shown as a schematic in Fig. 3.

There are many cases where observation of a cross section is important and necessary. SEM observation of flexure creep tested specimens is one of those cases. The bending moment creates a stress gradient in the specimen and cross section of bending specimen is exposed to both tensile and compressive stress as it is seen in Fig. 3. Therefore the deformation or strain is different at tensile and compressive regions.

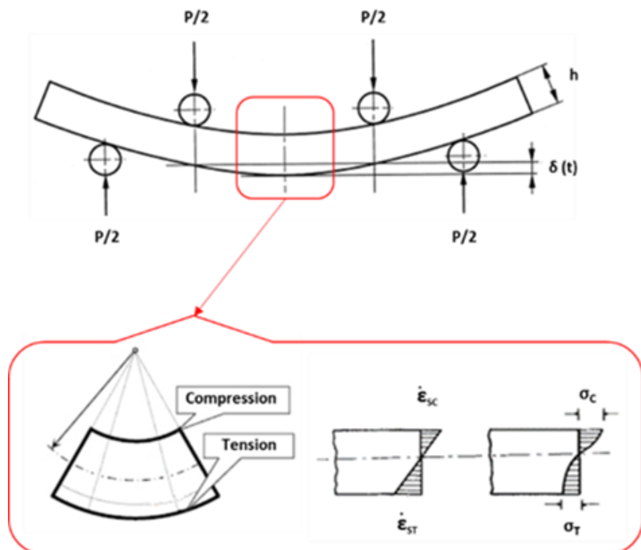

Fig. 3. *The loading configuration, deformed shape, stress and strain distribution occurring in bending testing*

The deformation or strain rates of Silicon Nitride based ceramics depends on loading configurations such as tension and compression. Strain rates in tension are more than in compression. Since creep damage occurs in the tensile side of a bend specimen, it necessary to observe cross section of crept bending specimen for SEM analysis [4, 12, 13]. Because of that, cross section area in the middle of specimen was determined area of interest (AOI) for sectioning as it seen Fig 4.

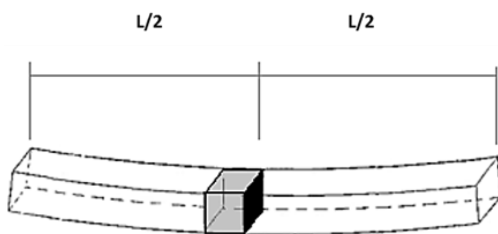

Fig. 4. *Determined area of interest (AOI) for sectioning*

The AOI section were cut and then prepared for the cross sectional SEM observation by CP method. Before locating of AOI section on cross polisher, the AOI section were mounted and ground to get a fresh surface and to reduce damage occurred while cutting.

C. *Cross Polishing (CP) Method*

Different cross section sample preparation methods such as mechanical polishing, microtome, Focused Ion Beam (FIB), and Cross Polishing (CP) can be used for microstructural analysis. In this study, cross section polishing of AOI section was performed by a cross section polisher (Jeol SM-090110) (Fig. 5). After grinding, AOI section were removed from the mold and then cross section polishing was performed by use of the cross polisher.

Fig. 5. *Cross section polisher (Jeol SM-090110)*

Cross section polisher has an argon ion beam gun and a chamber with vacuum capability. The specimen is located on a holder and a shielding plate in that chamber (Fig. 6). The region to be cross polished is chosen by an optical microscope. After vacuuming, selected region is exposed to argon ion beam with a determined time. Therefore, the section that is not covered by the shielding plate irradiated with argon ions (Fig. 7).

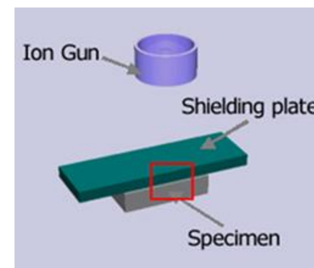

Fig. 6. *Schematic diagram of the cross polisher [14]*

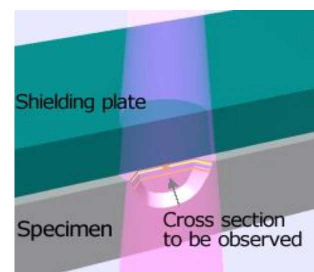

Fig. 7. *Schematic diagram during processing [14]*

III. Results and Discussions

The microstructures of cross section polished crept sample was investigated by using scanning electron microscope (SEM-ZEISS SUPRA 50 VP) attached

with an energy dispersive X-ray spectrometer (EDX Oxford Inca) (Fig. 8).

The general microstructure of sintered SiAlON composite ceramic investigated consists of α-SiAlON, β-SiAlON grains, and secondary phases. These phases can be distinguished on backscattered electron (BE) image, since these phases contain elements with different atomic number. Since heavy elements (high atomic number) backscatter electrons more strongly than light elements (low atomic number), and thus appear brighter in the image, BSE are used to detect contrast between areas with different chemical compositions.

Fig. 8. *Scanning Electron Microscope*

β-SiAlON grains are black colored and more needle like, whereas α-SiAlON grains are grey colored and more equiaxed whilst secondary phases appear fine grained and white colored, because of their increasing rare earth element content respectively [15]. The backscattered electron (BE) images of cross section polished crept α/β-SiAlON composite ceramic material at different magnifications are demonstrated in the figures which are numbered between 9 and 11.

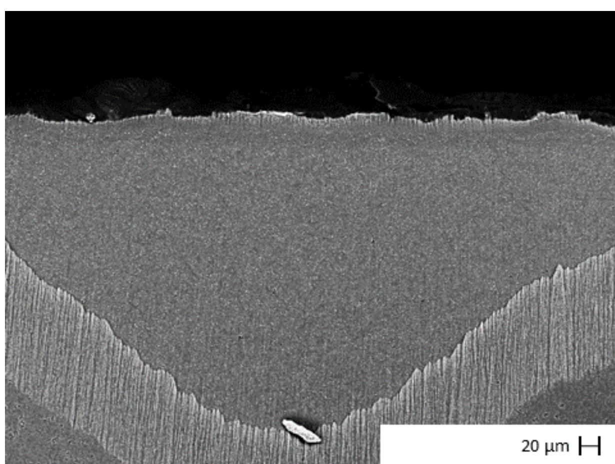

Fig. 9. *Back-scattered SEM image of a cross section of the crept specimen (500 x)*

Fig. 9 demonstrates general view of the cross section polished section cut from the crept sample. The "V" shaped region across bottom of image occurred by argon ion milling under effect of shielding plate during cross section polishing. The region above that "V" shaped region is AOI (area of interest) section at tensile side of crept bending specimen. The cross section polished part of AOI imaged using the BE signal clearly shows the constituent creep and oxidation damage zones.

Several sub-layer shaped damage zones resulted from creep and oxidation can be visually distinguished on AOI in Fig. 10. There is an unaffected zone containing α-SiAlON and β-SiAlON grains along with secondary phases at the bottom of BE image (Fig. 10). This unaffected zone is shown more clearly in Fig 11). There is a layer of oxidation scale on the top of BE image, and under of it there is a depleted zone which contains less secondary phase and more porosities than the unaffected zone.

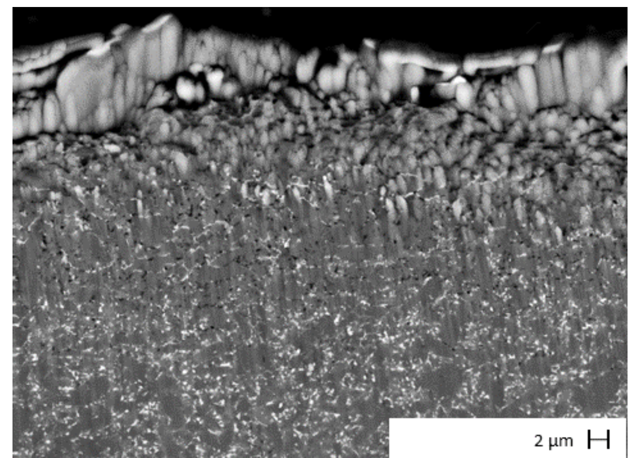

Fig. 10. *Back-scattered SEM image of a cross section of the crept specimen (5000 x)*

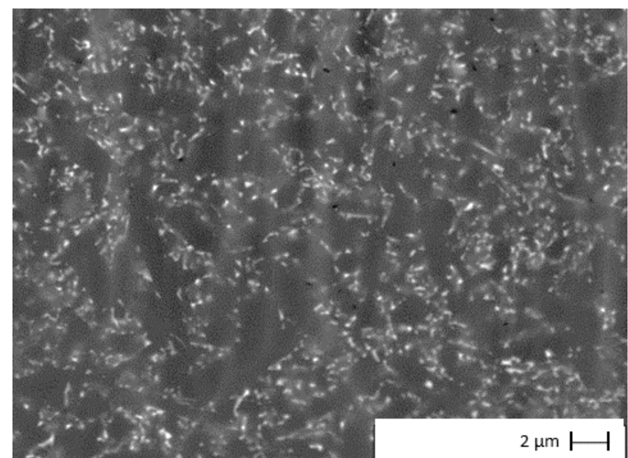

Fig. 11. *Back-scattered SEM image of a cross section of the crept specimen (10000 x)*

IV. Conclusions

Since creep damage occurs in the tensile side of a bend specimen, it necessary to observe cross section of crept bending specimen for SEM analysis. Different cross section sample preparation methods such as mechanical polishing, microtome, Focused Ion Beam (FIB), and Cross Polishing (CP) can be used for microstructural analysis. In this study, microstructure of

crept ceramics prepared by cross section polishing method was investigated by using SEM.

It is known that well-polished surface is required for Scanning Electron Microscope (SEM) analysis to accomplish successful observation. Any damage such as scratches or pull-outs, which might occur via mechanically polishing methods, was not observed. All of the phases such as α-SiAlON, β-SiAlON and secondary phases were observed by SEM analysis. The creep and oxidation damage zones were observed successfully too.

It has found that cross section polishing method is very useful for general observation of creep and oxidation damage ceramics. However, more detailed microstructural analysis such as Transmission Electron Microscopy (TEM) analysis is required to determine active creep mechanism(s).

ACKNOWLEDGEMENT

I would like to thank MDA Advanced Ceramic Technology A.S¸. for supplying of α/β-SiAlON composites, Assistant of Prof. Hilmi Yurdakul and for their help on microscopy investigations and Prof. Dr. Servet Turan and Associate of Prof. Dilek TURAN for useful discussions.

REFERENCES

[1] U. Taffner, V. Carle, U. Schafer and M. J. Hoffmann, “Preparation and microstructural analysis of high-performance ceramics”, ASM Handbook Volume 9: Metallography and Microstructures, ASM International, 2004.

[2] M. H. Lewis, and R. S. Dobedoe, “Creep of ceramics”, Encyclopedia of Materials: Science and Technology, 2002.

[3] W. D. Callister and D. G. Rethwisch, “Materials Science and Engineering: An Introduction”, 9th Edition, 2014.

[4] K. M. Fox and J. R. Hellmann, “Microstructure and creep behavior of silicon nitride and SiAlONs”, Int. J. Appl. Ceram. Technol. 5, 2008, pp. 138–154.

[5] Choi, S. R. and Holland, F. A., “Silicon nitride creep under various specimen-loading configurations”, NASA/TM-210026, 2000.

[6] D. N. Leonard, G. W. Chandler, and S. Seraphin, “Scanning electron microscopy, Characterization of Materials, 2012.

[7] A. Uludag, “Microstructural effect on creep of SiAlON ceramics”, Journal of Multidisciplinary Engineering Science and Technology), Vol. 2 Issue 5, 2015.

[8] M. Shibata, “Cross section specimen preparation device using argon ion beam for SEM”, Jeol News.

[9] http://www.nanoscience.com/technology/sem-technology/sample-electron-interaction/ accessed on November 22, 2016.

[10] S. Hampshire, “Silicon nitride ceramics – review of structure, processing and properties”, Journal of Achievements in Materials and Manufacturing Engineering, 24, 2007.

[11] P. E. Stutzman and J. R. Clifton, “Specimen preparation for scanning electron microscopy”, Proceedings of the Twenty-First International Conference on Cement Microscopy, L. Jany and A. Nisperos, Eds., 1999, Nevada, USA, pp. 10-22.

[12] C. F. Chen and T. Chuang, "Improved analysis for flexural creep with application to Sialon ceramics." Journal of the American Ceramic Society 73(8): 2366-2373, 1990.

[13] W. E. Luecke, S. M. Wiederhorn, B. J. Hockey, R. F. J. Krause, and G. G. Long, ‘‘Cavitation contributes substantially to tensile creep in Silicon Nitride,’’ J. Am. Ceram. Soc., 78 [8] 2085–96, 1995.

[14] http://www.jeol.co.jp/en/science/cp.html accessed on November 22, 2016.

[15] A. Uludag and D. Turan, “High temperature bending creep behavior of a multi-cation doped α/β-SiAlON composite”, Ceramics International 37 921–926, 2011.