Compressive Creep Deformation Test Using a Spark Plasma Sintering (SPS) Apparatus

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1. Introduction

Several studies [1-3] reported that creep of the ceramics was the main mechanism occurring in the densification final stage during pressure-assisted sintering. Therefore, during the high temperature sintering experiments using Spark Plasma Sintering (SPS), creep parameters were noted and analysed. These parameters were in good agreement with those obtained from conventional creep experiments.

For that reason, Spark Plasma Sintering (SPS) has recently been employed as an accurate high-temperature creep testing system through a special configuration [1,4,5]. This configuration consists of outer graphite dies and inner dies made of Yttiria-Stabilized Zirconia (YSZ) or Alumina (Al₂O₃) that work as insulators in addition to silicon carbide (SiC) that acts as a supporting material. Such an apparatus provides accurate output data, including temperature, pressure, and relative punch displacement (RPD) as well as electric field parameters.

Based on recent studies [1,4,5] that utilized this configuration, the setup was capable to be used for creep tests of metals and ceramics. The results, extrapolated to higher temperatures and lower stresses, were in good agreement with data reported in the literature.

One of the main disadvantages of such an apparatus is that a constant strain rate cannot be set as a parameter. High temperature compressive strength cannot be investigated. But in case of creep, tests are typically performed under a constant stress or load, with the test beginning only after a mechanical equilibrium, in which all thermal expansions and deflections of the system are already finished, is reached.

In this study, the procedure of performing uniaxial compressive creep tests using an SPS apparatus is discussed. The capability of SPS to be used as a creep test system should contribute to measuring the creep behaviors of new metallic or ceramic materials. In that regard, it will be used to obtain creep data of ceramic materials that will be used in advanced nuclear fuel designs and in the development of various advanced materials.

2. Experimental Procedure

To conduct creep tests using SPS, several specific features of the apparatus also have to be considered. For example, the minimal force that has to be applied so that the SPS system can operate needs a certain minimal cross-section of the specimens that determines a desired testing pressure.

Another concern when conducting the creep tests is the effect of an electromagnetic field within the SPS apparatus. Generally, a relatively low voltage (≤ 10 V) is applied to the whole setup, while a very high electric current is employed (1–10 kA) [1]. For conductive materials, the high current significantly affects creep behavior, with this being attributed to the effects of electro-plasticity. Therefore, an isolating material can be used between the testing sample and the mold punches to avoid passing the current through the sample. Al2O3 is one example of an isolating material that can be used.

Fig. 1 shows a schematic image of the SPS setup. A sample is placed between the graphite punches of the apparatus. A thermocouple or a pyrometer can be used for the temperature measurements.



Fig. 1. The SPS setup for the creep test.

Temperature, pressure (force), and displacement are the parameters of the major significance. The displacement results can be analyzed to calculate the strain and the creep strain rate. Repeating the experiment at different temperatures and stresses leads to the calculations of the activation energy and the stress component, respectively, according to the following equation [1,4,5]:

$$\dot{\varepsilon} = A\sigma^n \exp\left(\frac{-Q}{RT}\right) \dots (1)$$

where $\dot{\epsilon}$ is the creep rate, A is the creep constant, σ is the applied load, n is the stress exponent, Q is the activation energy, R is the gas constant and T is the temperature.

It is worth mentioning that in the beginning of the experiment, the sample experiences thermal expansion and the SPS Relative Punch Displacement (RPD) reading gives a negative value while the sample pressure increases. After reaching a certain temperature during the heating, the punches relative displacement reading starts to increase and the pressure reading decreases as the sample starts to deform. Therefore, the thermal expansion effect should be taken into account in the strain and strain rate calculations. For this reason, the minimum RPD reading is considered point zero in the displacement and the strain curves.

3. Published Results of Creep Tests Using SPS

Following the prescribed procedure, some published studies have conducted creep tests using SPS and obtained results that have good agreement with other data reported in the literature. An example of such studies is by Barak Ratzker et al. [1] who conducted creep tests using SPS on fine-grained alumina. Fig. 2 shows the creep strain rate plotted against strain for high and lower temperature ranges. In addition, Fig. 3 shows the creep strain rate versus stress of the conducted tests compared with the data previously published in the literature. The results show a good agreement with the data in the literature.



Fig. 2. Creep strain rate plotted against the strain, (a) high temperature and (b) lower temperature range [1].



Fig. 3. Strain rate versus stress for multiple studies of fine-grain alumina at various temperatures [1].

4. Conclusion

In this study, the procedure of the possibility of performing uniaxial compressive creep tests using an SPS apparatus is discussed. The concerns of using this apparatus for creep test seem to be easily overcome and obtained results show good agreement with the data published in the literature.

Evaluating the creep performance data from SPS for ceramics will give access to obtaining such missing properties of some ceramic materials that will be used in advanced nuclear fuel designs. For our future work, this apparatus will be used to evaluate the creep behavior of MoSiB alloys with different compositions and then compare the obtained results with the available published data in the literature.

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