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Creep and tensile tests on refractory metals at extremely high temperatures

Bernd Fischer [a], Stefan Vorberg [a], Rainer Völkl [b], Manuel Beschliesser [c,*], Andreas Hoffmann [c]

[a] *FH Jena—University of Applied Sciences, Jena, Germany*
[b] *University Bayreuth, Germany*
[c] *PLANSEE Aktiengesellschaft, Technology Centre, 6600 Reutte, Austria*

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Abstract

The need for mechanical properties at elevated temperatures is high for finite element modelling, process optimization, research and development or quality assurance purposes. Obtaining of this data is difficult, for refractory materials such as molybdenum or tungsten reliable data including precise strain measurement is required up to 2500 °C.

Over the last years a cooperation between PLANSEE and the University of Applied Sciences, Jena, Germany was built up. Within the internal research program "Basisdaten" (basic materials data) creep data and mechanical properties of molybdenum, tungsten and their alloys at very high temperatures could be achieved.

After a description of the unique test equipment at the University of Applied Sciences, Jena, the results of creep and tensile tests on molybdenum and tungsten sheet material are presented.

Keywords: Mechanical testing; Non-contact strain measurement; Tensile strength; Creep strength

1. Introduction

In the field of high-temperature applications, an increasing demand can be observed for the use of refractory metals as engineering materials. Therefore, a reliable determination of the high-temperature mechanical properties is essential. The high-temperature mechanical properties are required for the development of optimized alloys and processes, specifications in design engineering, modelling of component performance in industrial application and for quality assurance in production.

In many cases no data for very high temperatures is available and calculations and constructions are performed on the basis of experience and knowledge. With that no reliability and safety calculations can be performed. For research and development activities improvements in properties cannot be captured on a quantitative basis.

Because no commercial measuring system exists for creep and tensile tests conducted on metals at temperatures up to 3000 °C, special devices were developed and constructed at the University of Applied Sciences in Jena, Germany. The samples are heated directly by an electric current. Temperature measurement and control are achieved contact-free by a pyrometer and a PID-controller with a temperature tolerance of ±5 K. The strain measurement is also accomplished contact-free by a high resolution CCD-camera. Obtained data then are analyzed by the image processing program "*SuperCreep*" which has also been developed at the University of Applied Sciences. Results gained from these devices have been transferred to industrial partners over the last years which gave proof of the reliability of this testing equipment.

* Corresponding author. Tel.: +43 5672 600 2766; fax: +43 5672 600 536.
E-mail address: manuel.beschliesser@plansee.com (M. Beschliesser).

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In cooperation with PLANSEE uniaxial creep and tensile tests on sheet material of molybdenum, tungsten and their alloys which were taken from the commercial production line of PLANSEE have been performed.

The first part of this paper covers tensile tests of tungsten sheet material (thickness 1 mm). These tests should give answer to the question whether the results obtained in Jena fit to the results obtained at PLANSEE. Therefore, tensile tests on a sheet of the same batch were performed in Jena (testing temperature 1800–2500 °C) and in the Testing Laboratories in the Technology Centre of PLANSEE (testing temperature 1400–2100 °C).

The second part investigates the creep performance of Mo and ML (molybdenum based material, doped with La_2O_3 particles) sheet material (thickness 2 mm) at 1400 °C and 1600 °C up to 200 h.

2. Experimental

2.1. General design of test facilities at the University of Applied Sciences, Jena

The interest of the research group in Jena is focused on metallic materials for ultra-high-temperature applications. Envisaged test temperatures range up to 3000 °C for Re/W alloys [1]. Commercial test facilities either do not have the required specifications or are too expensive. This is the reason why these test facilities [2–6] were designed and built at the University of Applied Sciences, Jena.

Ohmic heating was chosen for easy access to the sample, fast heating and cooling cycles, and simplicity in design and operation. Low temperatures at the grips allow the use of inexpensive copper without the need for an active cooling system. Usually specimens in the form of thin wires or strips are tested. The test facilities permit tests either in air or under a protective gas atmosphere. All functions are computer controlled with the software *LabView* and *SuperCreep* [4–6], the later developed at the University of Applied Sciences, Jena for strain measurements by means of digital image analysis.

Both constant tensile load creep tests and high-temperature tensile tests can be executed. For constant engineering stress creep tests, the load is applied to the sample through a steel pull rod by means of calibrated weights. Alternatively, the specimen chamber can be mounted in a commercial test machine. The steel pull rod is then connected with the load cell at the crosshead of the test machine. A schematic diagram of the test facilities is given in Fig. 1.

The temperature is measured with a pyrometer. An adjustable response time down to 1 ms guarantees secure temperature control at high heating rates. A problem often encountered in pyrometry is that the spectral emissivity of the investigated material has to be known as a function of temperature, time and wavelength. Pt/Rh alloys show very slow oxidation at low temperatures. At temperatures above about 1000 °C their oxide scales evaporate. Neuer et al. [7,8] therefore recommend Pt/Rh alloys as reference materials.

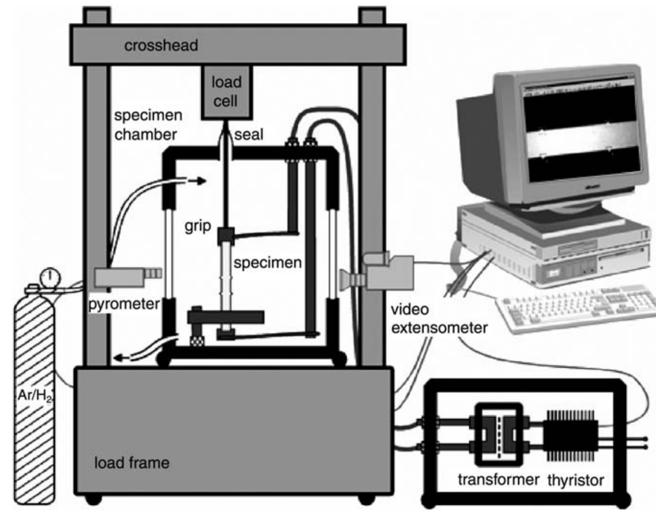

Fig. 1. Test facility to measure tensile and creep properties of metallic materials at temperatures up to 3000 °C.

Hence simple calibration of the test system for materials with unknown emissivity can be performed with a thin foil of a Pt/Rh alloy pasted on the specimen.

Strain is measured with a video extensometer controlled by the software *SuperCreep*. A variable exposure time of the CCD camera from 1 to 1000 ms allows images to be grabbed up to 2000 °C without introducing filters in the optical path. Telecentric lenses are used to avoid perspective distortions. *SuperCreep* continuously determines the distances between corresponding markers in the central zone of the specimen where the temperature is uniform. Suitable markers for high temperature tests can be made by laser cutting samples with small shoulders from the sheet material (Fig. 2a), or simply by winding thin wires around the specimen (Fig. 2b).

2.2. Performance of test facilities

The temperature distribution was measured by a second pyrometer on a 100 mm long strip of a Pt–10% Rh alloy [5]. The maximum temperature was 1500 °C at the specimen centre. In a zone 30 mm around the specimen centre the temperature was 1500 ± 5 °C. In a zone 10 mm between the markers the temperature was within 1500 ± 2 °C. During a following creep test the temperature between the markers could be held to 1500 ± 3 °C until necking occurred. Due to ohmic heating the temperature outside a necked region is always lower, whereas in the necked region the temperature is kept at the desired value.

The test facility allows fast heating and cooling cycles. A maximum temperature of 1506 °C at the centre of the Pt–10% Rh DPH strip was reached 12 s after turning on power. After 15 s the maximum temperature was held at 1500 ± 1 °C. After switching off the power, the specimen took about 20 s to cool down to 750 °C. Heating and cooling rates of +100 °C/s and −30 °C/s, respectively, can be reached [5].