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In-situ fatigue in an environmental scanning electron microscope – Potential and current limitations

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Abstract

The present study provides a survey on *in situ* fatigue devices suitable for loading specimens in a scanning electron microscope (SEM). Particular emphasis is placed on the experimental methods employed to operate a small-scale load frame in an environmental SEM (ESEM). Specimen design and surface preparation, various modes of conducting the fatigue tests, specimen heating and imaging at elevated temperatures are considered.

In addition, recent data from *in situ* fatigue studies conducted in an ESEM between room temperature and 600 °C are summarized. Fatigue tests conducted on AM60B cast magnesium demonstrate a substantial environmental effect on cyclic deformation and fatigue damage evolution even at room temperature. Fatigue behaviour of high-temperature titanium alloy IMI 834 at 400 °C illustrates a completely different effect of environment on slip band formation and crack nucleation as compared to AM60B. Investigations performed on IMI 834 at 600 °C reveal current limitations of the *in situ* fatigue technique applied. An approach to overcome these limitations is discussed.

Ambient conditions can be simulated in an ESEM using pure water vapour atmosphere at a pressure similar to the partial pressure of the ambient environment. This is demonstrated for AM60B at room temperature and IMI 834 at 400 °C. However, this approach cannot necessarily be applied to all other material/environment combinations as is exemplarily shown for *in situ* fatigue loading of IMI 834 at 600 °C. Data obtained from *in situ* fatigue testing are compared to those from *post-mortem* SEM studies of failed specimens, which portray a wrong impression in certain cases. Experimental issues specific to environmental *in situ* testing at elevated temperatures are addressed as are the ramifications of such tests with respect to modelling of fatigue life.

Keywords: *In situ* fatigue; Cast magnesium; AM60B; High-temperature titanium alloy; IMI 834; Environmental degradation; Fatigue mechanisms; Microstructure; Crack initiation; Small crack growth; Damage evolution

1. Introduction

Accurate prediction of fatigue performance is a key issue in the design of many high performance technical components. The ever increasing demand for higher efficiency and light weight typical of power generation, aerospace and automotive industries calls for maximum exploitation of a material's properties. Thus, purely empirical models that heavily rely on large safety factors are of limited use. It is now widely recognized that models that are closely related to the microstructural processes provide a more reliable basis for life prediction, provided that the relevant microstructural damage mechanisms are accurately accounted for. Hence, there is increasing interest in fatigue tests combined with high-resolution microscopic techniques.

Dating back to the seventies of the last century, the potential of *in situ* fatigue studies conducted in a scanning electron microscope (SEM) has been realized as SEMs combine high lateral resolution and large depth of field

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with analytical capabilities [1–6]. Since that time, numerous studies have demonstrated that *in situ* fatigue in the SEM is an effective tool for the investigation of microstructural effects on the development of slip features [1,2,7–13] and on crack initiation [1,2,7,10–21], for the analysis of the mechanisms of crack growth [6,22–32], for the quantitative determination of small crack growth rates [14,24,28,30,33–39] and crack and crack tip opening displacements [5,23–26,30,34,36,38,40–42], for the examination of interactions between fatigue crack growth and the microstructure [4,14,16,18–21,24,35,37,39,43–58] as well as for the inspection of crack closure [5,22,31,33,34,41,59]. Recently, a node control mechanism has been introduced that allows a point of interest to remain within view with an accuracy of 1 μm [60].

The major limitations of most of those studies were imposed by the fact that the experiments had to be conducted at room temperature in a high-vacuum, whereas most components typically operate in a gaseous environment, which can simultaneously be combined with high-temperature conditions. In an overview on SEM cyclic loading stages operating world-wide in 1996, Davidson stated that there is a strong need to perform *in situ* experiments under the extremes of both temperature and environment [61]. By now, several *in situ* fatigue stages have built up various research, which allow for isothermal investigations in a high vacuum with maximum testing temperatures varying from 550 to 850 °C [10,14,17,24,35,41,54]. Thermal fatigue has also been examined under high-vacuum conditions [15]. However, an effect of environment on crack growth rate has been observed already at room temperature [24]. As environmental degradation usually is more pronounced at high temperatures, additional *ex situ* fatigue studies are commonly employed to verify the validity of the results from the *in situ* fatigue tests conducted in high vacuum.

With the advent of environmental scanning electron microscopes (ESEM) the situation has changed considerably [62,63]. Today's ESEMs typically allow for imaging in various gaseous environments at a pressure up to about 20–30 Torr, and thus, high-resolution data may be obtained under conditions relevant to those of actual components. Most procedures required for *in situ* fatigue testing conducted at room temperature in an ESEM are not much different from those required for tests in vacuum [18–21,31,37,39]. However, *in situ* fatigue studies under environmental high-temperature fatigue conditions are extremely challenging, and thus, such work is still extremely rare [11,12]. Given this scenario, the objectives of the present paper are to provide a detailed discussion on the experimental issues involved in room and high-temperature *in situ* fatigue studies, highlight the potential of the technique, but also to address current limitations and pitfalls to avoid. Environmental *in situ* fatigue testing at elevated temperatures is still in its infancy, and it is hoped that the current study will stimulate further research in this area.

In the following, room temperature tests conducted on a cast magnesium alloy are addressed first, as these demonstrate that substantial environmental effects on fatigue damage evolution can be observed in certain materials even at room temperature. The second part of the paper focuses on high-temperature *in situ* testing, and the examples provided are from recent fatigue tests on high-temperature titanium alloy IMI 834. Currently, the *in situ* fatigue tests cover the range between room temperature and 600 °C.

2. Experimental details

2.1. ESEM, mechanical test system

A Philips XL 40 ESEM was chosen as it provides a chamber large enough to accommodate the small-scale load frame used in the present study. Typically, the ESEM can be operated in gaseous environments with a pressure up to $p = 2.7 \times 10^3$ Pa (20 Torr) or in a high-vacuum mode at $p < 10^{-3}$ Pa (7.4×10^{-6} Torr). In fatigue tests where water vapour was used as the testing environment, a pressure of 20 and 12 Torr corresponds to the partial pressure of water vapour at 100% and 60% relative humidity at ambient room temperature conditions, respectively. Fig. 1 shows an overview of the small-scale screw-driven load frame including load cell (1), displacement transducer (2) and extensometer (3) custom built by Kammrath + Weiss GmbH for the *in situ* fatigue studies in the ESEM. Maximum load capacity of the frame is 10 kN in both push and pull direction. The extensometer (3) seen in Fig. 1 below centre employs ceramic extension rods (4) and has a gauge length of 10 mm, which puts some constraint on the specimen geometry.

For high-temperature *in situ* fatigue studies in a SEM, specimen cross section is another important issue, as there are various conflicting issues that need to be addressed. Firstly, the specimens should have a thin gauge section in order to minimize the temperature gradient. The heating system currently employed allows for specimen temperatures up to 750 °C. With specimens having a thickness in

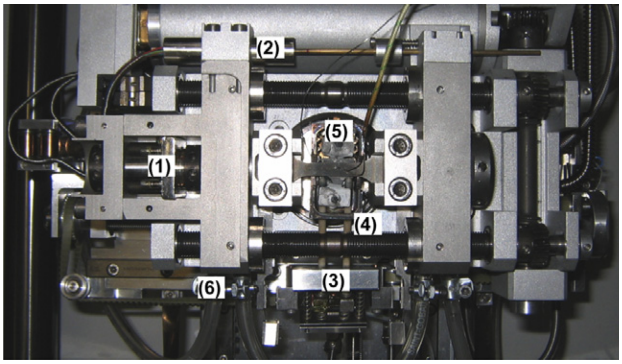

Fig. 1. Miniature load frame used for *in situ* fatigue testing in the ESEM (the heat shield that covers most of the specimen and the furnace has been removed). See main text for details.