LOW CYCLE FATIGUE BEHAVIOR OF POWDER METALLURGY STAINLESS STEEL/MG-PSZ COMPOSITE MATERIALS

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Abstract
This research work presents the low cycle fatigue behavior of powder metallurgy stainless steel/Mg-PSZ composite materials. The steel matrix is made of a conventional AISI 304 stainless steel. The ceramic reinforcement consists of MgO (~8 mol%) partially stabilized zirconia (Mg-PSZ). The unreinforced steel and composite materials containing 5 vol% and 10 vol% Mg-PSZ, respectively, were produced using hot pressing. The different materials were studied under total strain control at different strain amplitudes in order to clarify the influence of the ceramic reinforcement on the low cycle fatigue behavior. Microstructure investigations were performed to study the resulting deformation structures within the steel matrix and ceramic reinforcement, respectively.

1 Introduction
Particle-reinforced metal matrix composites are viewed with great interest for many structural applications because they combine the positive mechanical properties of the single components. Most studies were carried out on particle-reinforced lightweight-metals, since the resulting composites are characterized by improved properties, e.g. higher strength, stiffness and weight savings, compared to the non-reinforced metals [1].
A lot of work was done in the field of the fatigue behavior of MMC systems based on lightweight-metals such as aluminum or titanium matrix. Generally, it is stated that the fatigue behavior of MMC systems depends predominantly on the strength and microstructure of the metallic matrix [2-4], volume fraction, morphology, and size of the ceramic reinforcement [4-6] as well as on the control mode of the cyclic deformation tests [7]. In strain controlled low cycle fatigue tests at high strain amplitudes, the MMCs are characterized by shorter fatigue life times compared to the unreinforced matrix alloys [2, 5]. Moreover, the fatigue lives decrease significantly with increasing volume fraction of the reinforcing phase [2]. Conversely, in the high cycle fatigue regime under stress control the addition of a ceramic reinforcement can improve the fatigue life time [6, 8].
However, MMC systems based on a steel matrix have so far not been investigated in depth compared to the MMCs based on lightweight-metals. Moreover, using TRIP steels as metallic matrix and partially stabilized zirconia as ceramic reinforcement delivers the possibility of

producing MMCs which can show in both components phase transformations as a result of a plastic deformation or stress. Guo et al. [9] investigated the static and dynamic deformation behavior of 2Y-PSZ/TRIP steel composites. Recently, in the collaborative research center "TRIP-Matrix Composite", MMC systems based on high-alloyed TRIP steels and partially stabilized zirconia were studied [10-13]. As matrix material high-alloyed austenitic CrMnNi-steels are used which can show a transformation into ε and/or α'-martensite. The ceramic reinforcing phase consists of MgO partially stabilized zirconia (Mg-PSZ). Generally, zirconia can exist in three different modifications, i.e. in the cubic, tetragonal, or monoclinic modification, whereas the monoclinic phase is the stable modification at room temperature [14]. Using MgO as stabilizer allows conserving the tetragonal phase in metastable conditions at room temperature. As a result of an external load the tetragonal phase can transform in a stress-assisted way into the monoclinic modification resulting in a reasonable toughening effect [15].

In a recent work of the authors [16] the cyclic deformation behavior of comparable MMCs was investigated which have been produced using the Spark Plasma Sintering technique. The present study investigates the low cycle fatigue behavior of MMCs which have been produced by a different production route using hot pressing.

2 Materials and testing methods

As steel matrix a conventional AISI 304 powder (TLS, Bitterfeld, Germany) was used, cf. Table 1. As reported in literature, this steel grade can show a martensitic transformation in strain controlled fatigue tests [17]. However, the presently used steel powder has a relatively high nitrogen content and thus an increased austenite stability hindering the martensitic transformation. Therefore, a pronounced martensitic transformation was not expected. Moreover, as a result of the powder metallurgy approach the powder contains impurities of Al_2O_3 in the order of approximately 3 vol%.

AISI 304	C	Ni	Cr	Mn	Si	Mo	N	Fe
wt%	0.06	8.6	17.9	1.9	0.4	0.4	0.14	bal.

Table 1. Chemical composition of the AISI 304 steel powder.

Mg-PSZ	MgO	HfO_2	Al_2O_3	SiO_2	CaO	TiO_2	Fe_2O_3	Na_2O	ZrO_2
wt%	2.82	1.74	0.38	0.41	0.15	0.13	0.13	0.10	bal.

Table 2. Chemical composition of the Mg-PSZ powder.

A commercially available 3 wt% (~8 mol%) MgO partially stabilized zirconia powder (Saint-Gobain, USA) was used as reinforcing ceramic phase, Table 2. The steel powder was mixed for 1 h with 5 and 10 vol% of the Mg-PSZ, respectively, using a ball mill. Discs with a diameter of 150 mm were precompressed at 60 MPa. Subsequently, the discs were sintered using the hot pressing technique under vacuum conditions. The precompressed discs were heated up to 1250 °C with a heating rate of 10 K/min, kept at this temperature for 30 minutes and cooled afterwards with a cooling rate of 5 K/min. During the dwell time a pressure of approximately 30 MPa was applied. In comparison, during the production using SPS [16] the maximum temperature was 1100 °C and the dwell time 5 minutes.

Figure 1 shows typical features of the Mg-PSZ microstructure in the as-sintered conditions. Two characteristic morphologies were observed. Evidently, a lot of grains contain laths which were identified as monoclinic phase [16], see Figure 1a. Moreover, some grains are characterized by an outer monoclinic fringe, Figure 1a and b. Both features are correlated with a partial destabilization of the Mg-PSZ during the sintering process which was observed also in previous research works [11, 12]. The stabilizer Mg diffuses into the steel matrix favouring a martensitic transformation to the monoclinic phase.

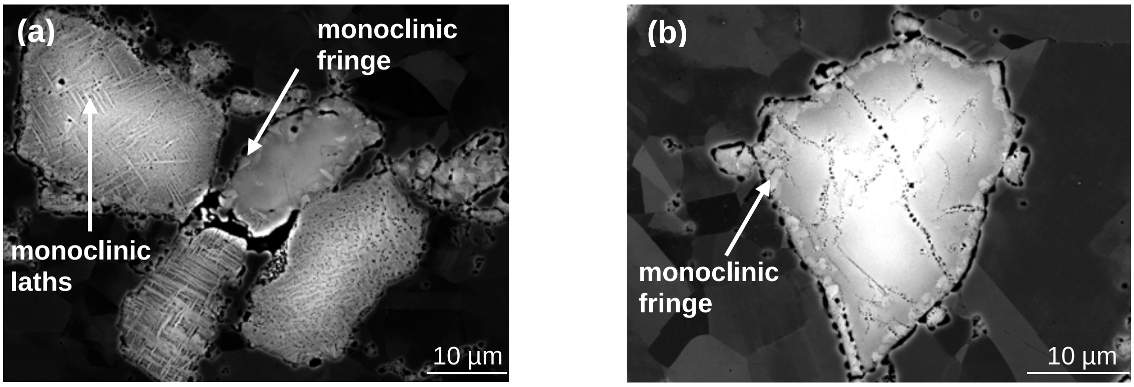

Figure 1. SEM observations in BSE contrast of the ceramic microstructures in the as-sintered conditions. (a) Mg-PSZ particles showing monoclinic laths and outer monoclinic fringe. (b) Mg-PSZ particle with an outer monoclinic fringe.

Total strain controlled low cycle fatigue tests were performed on a servohydraulic testing system MTS Landmark 250 (250 kN) using triangular load-time functions. Total strain amplitudes were varied between $2.5\times10^{-3} \leq \Delta\varepsilon_t/2 \leq 8\times10^{-3}$ at a constant strain ratio of $R_\varepsilon = -1$ and constant total strain rate of 4×10^{-3} s^{-1}. A clip-on extensometer was applied to measure the strain. Mechanically polished cylindrical specimens with a gauge diameter of 8 mm and a gauge length of 14 mm were used. Specimens for microstructure investigations were cut parallel to the loading axis. The sample preparation consisted of conventional polishing steps followed by final vibration polishing for 24 h. The microstructures were investigated using a field-emission scanning electron microscope Tescan Mira3 FE-SEM equipped with a retractable four-quadrant BSE (backscattered electron) detector.

3 Results and Discussion

3.1 Cyclic deformation behavior

Figure 2 shows the cyclic deformation curves of the unreinforced AISI 304 steel and MMC with 10 vol% Mg-PSZ at different total strain amplitudes. Both materials are characterized by an initial hardening followed by cyclic softening up to the final fracture. For a given total strain amplitude the addition of the Mg-PSZ ceramic reinforcement yields increasing stress

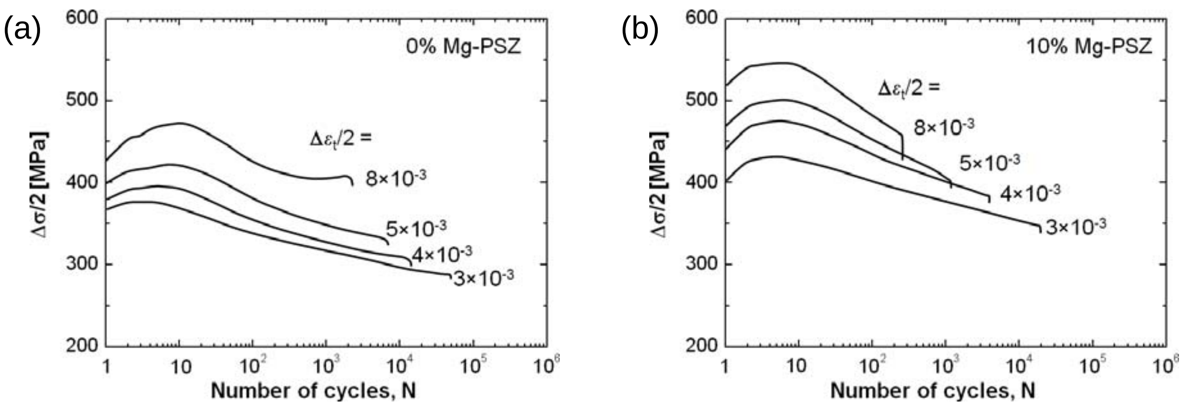

Figure 2. Cyclic deformation curves of the unreinforced AISI 304 steel (a) and MMC with 10 vol% Mg-PSZ (b).

amplitudes, $\Delta\sigma/2$. A pronounced secondary hardening cannot be observed and thus a significant deformation-induced martensitic transformation can be excluded.
Figure 3a compares the cyclic deformation behavior of the three investigated materials at a total strain amplitude of $\Delta\varepsilon_t/2 = 8\times10^{-3}$. The ceramic reinforcement results in a significant hardening effect which increases with increasing amount of Mg-PSZ. With increasing volume fraction of Mg-PSZ the fatigue life times are reduced. Moreover, in the MMCs more negative mean stresses are observed which indicate a more pronounced material damage in the MMCs. Already damaged areas in terms of broken or debonded particles cannot bear load under tensile stresses. Conversely, under compressive stresses the damaged areas are compressed without a significant loss of stiffness leading to negative mean stresses during cyclic loading [18].

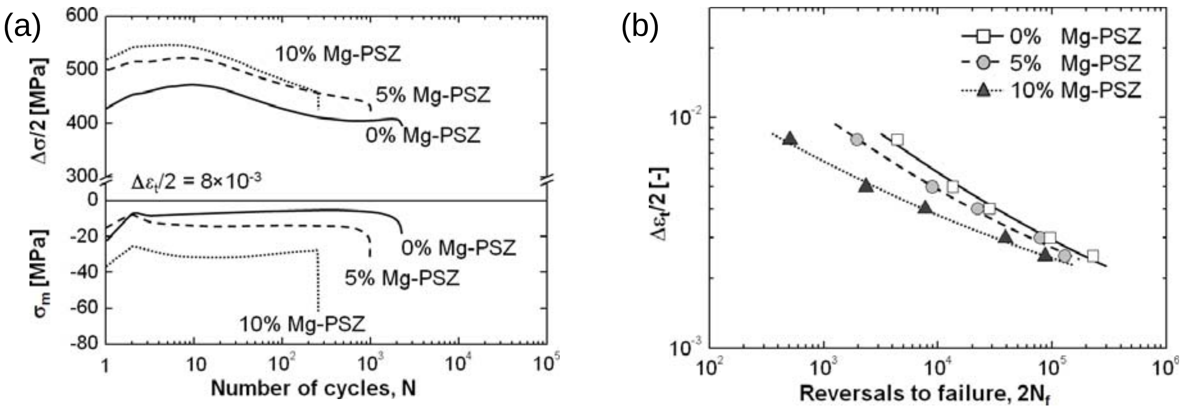

Figure 3. (a) Cyclic deformation curves and evolution of the mean stress (σ_m) of the unreinforced steel compared to the MMCs with 5 and 10 vol% Mg-PSZ, respectively, at a total strain amplitude of $\Delta\varepsilon_t/2=8\times10^{-3}$. (b) Fatigue lives of the unreinforced steel compared to the MMCs with 5 and 10 vol% Mg-PSZ in terms of total strain amplitudes $\Delta\varepsilon_t/2$ vs. number of reversals to failure ($2N_f$).

Figure 3b shows the correlation between the applied total strain amplitudes and the number of reversals to failure ($2N_f$). At all investigated total strain amplitudes the addition of Mg-PSZ results in a reduction of the fatigue lives. It is important to note, that the differences in the fatigue life time decrease with decreasing strain amplitudes. Thus, in the LCF regime a significant life time reduction compared to the unreinforced steel is observed, which correlates well with the findings of former research works on the low cycle fatigue behavior of MMCs [2, 5, 16]. Moreover, the fatigue life time reduction increases with an increasing amount of the Mg-PSZ reinforcement.
The fatigue life times of the presently studied materials are comparable with the fatigue lives of the recently investigated MMCs which have been produced using the spark plasma sintering technique [16]. Thus, in the investigated range of total strain amplitudes a significant influence of the production route in terms of hot pressing or spark plasma sintering can be excluded.
Alternatively, in Figure 4 the maximum stress amplitude $\Delta\sigma/2_{max}$ of each test and the stress amplitude at the half life time $\Delta\sigma/2(N_f/2)$ are plotted versus the fatigue life times ($2N_f$) of the unreinforced steel and the MMCs containing 5 and 10 vol% Mg-PSZ, respectively. In stress controlled tests these plots correspond to the well-known S-N-plot. In both figures it can be seen that the MMCs are characterized by higher fatigue life times at a given stress amplitude. Furthermore, the MMC with 5 vol% Mg-PSZ shows slightly higher fatigue lives compared to the MMC with 10 vol% Mg-PSZ. These results support the assumption that in the MMC with increased volume fraction of Mg-PSZ an accelerated and more pronounced material damage occurs.

Consequently, it can be assumed that in stress controlled tests, especially in the HCF regime, the MMCs show higher fatigue life times compared to the unreinforced steel. These findings correlate well with the observations of several research groups, which stated higher fatigue life times of MMCs in stress controlled tests in the HCF regime compared to the unreinforced metal matrix [6, 8].

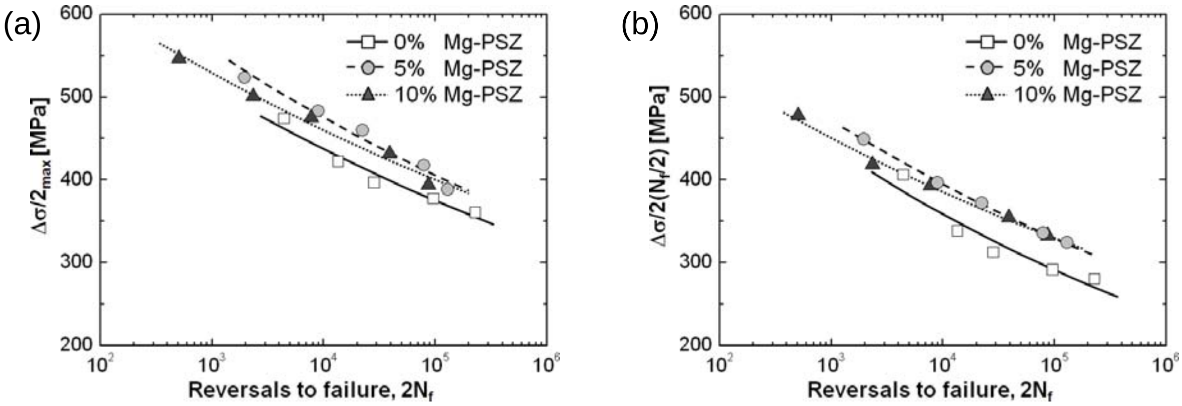

Figure 4. (a) Correlation between the maximum stress amplitude $\Delta\sigma/2_{max}$ and the number of reversals to failure ($2N_f$). (b) Stress amplitudes at the half life time $\Delta\sigma/2(N_f/2)$ vs. number of reversals to failure ($2N_f$).

3.2 Failure mechanisms

In order to clarify the failure mechanisms, detailed SEM analyses were performed on MMCs containing 10 vol% Mg-PSZ. The SEM figures in Figure 5 reveal that the commonly found failure mechanisms are observed, i.e. debonding of ceramic particles from the steel matrix, particle fracture and crack coalescence, respectively. Debonding as failure mechanism indicates a less pronounced interfacial bonding between the ceramic particles and the steel matrix. Particle fracturing can be predominantly observed in larger ceramic grains, which correlates well with the results of Li et al. [5]. These particles were broken perpendicular to the loading axis. As commonly stated, this failure mechanism is observed predominantly in MMCs under tensile loading and indicates a good interfacial bonding leading to a good load transfer to the ceramic reinforcement [19]. It can be assumed that the final fracture mechanism is given by the growth and coalescence of voids from broken and debonded ceramic particles. As stated by Poza et al. [19], this last stage in the fracture process is fast and starts when a critical amount of broken ceramic reinforcements is reached.

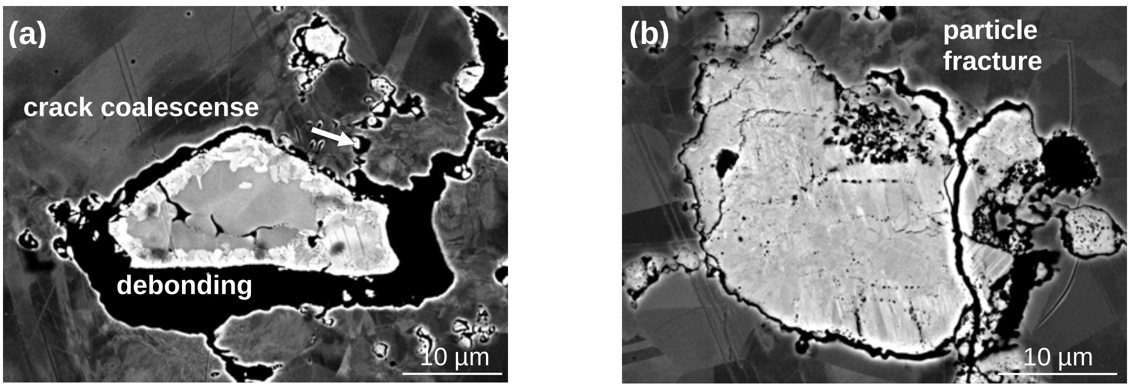

Figure 5. Deformation microstructures of the MMC containing 10 vol% Mg-PSZ. The sample was cyclically deformed at $\Delta\varepsilon_t/2=3\times10^{-3}$. Loading axis horizontal.

4 Conclusions

The scope of this study was to investigate the low cycle fatigue behavior of metal matrix composites consisting of AISI 304 steel as matrix material and Mg-PSZ as ceramic reinforcing phase, produced by hot pressing. The following main conclusions can be drawn:

(1) The low cycle fatigue behavior of the investigated materials is characterized by initial cyclic hardening followed by continuous cyclic softening up to the final fracture.
(2) The addition of Mg-PSZ results in a strong hardening effect under cyclic loading.
(3) In the studied total strain controlled fatigue tests, an increasing volume fraction of Mg-PSZ results in decreasing fatigue life times.
(4) The dominant damage mechanisms in the MMCs are debonding, particle fracture and crack coalescence.

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