CHARACTERIZATION AND IMPROVEMENT OF SMA-POLYMER INTERFACE IN ACTIVE HYBRID COMPOSITES

J. Jungbluth[1], C. Schmidt[2], A. Gapeeva[2], S. Bruns[3], F. Beckmann[4], J. Moosmann[4], Berit Zeller-Plumhoff[3], J. Carstensen[2], R. Adelung[2] and M. Gurka[1]

[1] Leibniz-Institut für Verbundwerkstoffe GmbH, Erwin-Schrödinger-Straße 58, 67663 Kaiserslautern,

[2] Christian-Albrechts-Universität zu Kiel, Department of Materials Science, Chair for Functional Nanomaterials, Faculty of Engineering, Kiel University, Kiel, Kaiserstraße 2, D-24143 Kiel, Germany

[3] Helmholtz-Zentrum Hereon, Institut für Metallische Biomaterialien, Max-Planck-Str.1, 21502 Geesthacht

[4] Helmholtz-Zentrum Hereon, Institut für Materialphysik, Max-Planck-Str.1, 21502 Geesthacht

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ABSTRACT

In this paper, we present a new approach to characterize the load transfer capability of the interface between surface-structured shape memory alloy (SMA) wires and the surrounding polymer epoxy matrix in an active hybrid composite (HC). We combined imaging methods, such as stress optics, synchrotron radiation-based micro computed tomography (µCT) and scanning electron microscopy (SEM) with mechanical pull-out tests to investigate the influence of the surface structure and increase in surface area on the mechanical load bearing capability of the interface in a quantitative manner. Structuring of the SMA wire surface was achieved by using selective electrochemical etching. With that, surface pits can be introduced into the SMA wire surface, resulting in an improvement of force transmission between SMA wire and surrounding polymer matrix due to surface enlargement and mechanical interlocking. The force transmission of three different SMA wire surface finishes - as-delivered - Structure 1 and Structure 2 was tested with the pull-out test and the force of first failure was compared. By that, an influence of the SMA wire surface morphology on the resulting force of first failure could be demonstrated. By using µCT for the surface analysis of the SMA wire, a quantitative analysis of the resulting SMA wire surface morphology can be correlated with the results of the mechanical pull-out test combined with stress optics.

1 INTRODUCTION

Active hybrid composites (HC) made from NiTi shape memory alloy (SMA) wires and a stiff fiber-reinforced polymer (FRP) represent a new class of functional materials. [1,2] SMA is characterized by a thermoelastic phase transformation, turning heat into mechanical work (up to 8% contraction at up to 150 MPa over a temperature change of dT = 20 K), resulting in an outstanding gravimetric energy density. In this way, new adjustable composite materials can be designed, which are interesting for various applications such as morphing surfaces in aviation or as fast and silent actuators. [2, 3]

However, thermal and mechanical load transmission from the SMA wire to the surrounding polymer matrix is the limiting factor, and a significant efforts have been made to understand and optimize the mechanisms at this interface. [4, 5, 6] The characterization of interface strength is usually done by using mechanical pull-out testing [7] and, measuring the maximum shear stress that can be transferred across the interface. Due to the often unknown boundary conditions, such as exact sample geometry and the free edge effect, results must be analysed and interpreted carefully. [4, 5] By using additional optical investigation methods such as 3D X-ray imaging or stress optics during mechanical pull-out testing, a deeper understanding of the failure process in the interface can be established. [4] CT analysis and SEM are used for detailed quantitative assessment of the SMA wire surface, while stress optics detects precisely the moment of first failure during the pull-out test in the interface. Often this cannot be seen in the stress-strain curve. [8]

For improvement of the force transfer capability, different approaches for modification of the SMA surface were proposed. [8, 9, 10, 11] To ensure a homogeneous force transmission at every point along the SMA wire length without additionally introduced force-transferring elements, such as clips, crimps or weldings on the SMA wire, selective electrochemical etching is a rather new process for SMA wire surface modification. [4] The SMA wire is structured over its entire length with surface pits (see figure 1). The generation of these pits on the surface of the SMA wire can be achieved with high precision in terms of their size, shape, and distribution through the regulation of electrochemical etching parameters, such as current densities, amplitude, and pulse duration. This results in a reproducible process for pit formation. Since this selective electrochemical etching process erodes preferably defects and nickel-rich areas from the SMA wire surface, the SMA wire surface quality is simultaneously enhanced. The combination of the selectively structured surface of the SMA wire and a low viscosity epoxy resin leads to an interlocking at the interface that significantly improves the force transmission between the polymer and the SMA wire. In [4], we demonstrated the benefits of this process regarding the force transmission from the SMA wire to the polymer by using the pull-out test. However, SMA wires with a diameter of 1 mm were used in this study. These are unusual for the applications described for HC. The transferability of the results to thinner SMA wires is part of this investigation. In addition, it has already been suggested in [4] that the surface morphology of the SMA wires has an influence on the type of interfacial failure. To verify this, two different structural characteristics were applied to the SMA wires. By using optical investigation methods, the introduced structure can be described and correlated with the data gathered with pull-out test combined with stress-optics.

2 MATERIAL

2.1 SMA WIRE

A NiTi one-way effect SMA wire Alloy H from Memry GmbH with a polished oxide-free surface finish was used. The SMA wire has a diameter of 0.5 mm. The surface selective etching process described in [4] was used to introduce etch pits (partly omega-shaped) into the SMA wire surface. With that, a reproducible and over the length of the SMA wire homogenous surface structure was introduced into the SMA wire surface. By changing parameters like current pulse duration and amplitude, the SMA surface structure can be controlled and different SMA wire surface morphologies can be introduced. The surface structuring is reproducible and does not affect the stress-strain characteristics of the SMA wire. [4] In this research, two different surface structure configurations were compared with the as-delivered non-treated SMA wire surface. A detailed optical characterization of the two structured SMA wire surfaces compared in this research is given by the SEM images of the SMA wire surfaces in *Figure 1*. A clear morphological difference between both configurations can obviously be seen. Structure 1 has a higher and more evenly distributed etch pits density smaller etch pits, more etch pits and near pits merged to larger structures. The surface of Structure 2 shows roundish formed pits homogenously distributed. The pit size is significantly larger compared to structure 1.

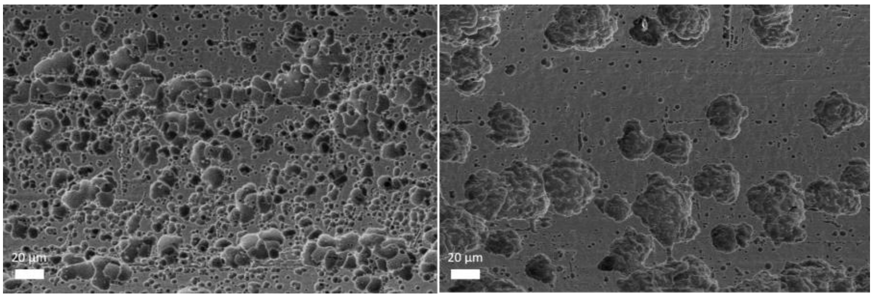

Figure 1: SEM images of the structured SMA wire surface treated with two different recipes, Structure 1 (left), Structure 2 (right)

2.2 MATRIX

A cold-curing, transparent epoxy resin was used for the SMA wire embedding in this research. The polymer, Araldite® LY 5052 (100g) and the hardener Aradur® 5052 (38g), purchased from Huntsman Corporation (The Woodlands, USA), were mixed by hand for two minutes. After that, a curing cycle of two days at room temperature with 9 bar pressure in an autoclave took place. Prior to testing, the specimens were cured at least for another five days in ambient environment at room temperature and atmospheric pressure. Important mechanical properties of the epoxy used are the ultimate elongation at break of 1.5-2.5 % and the tensile modulus of 3350 MPa. [12]

2.3 SPECIMEN PREPARATION

The specimen preparation procedure was performed in accordance with the methodology outlined in [4]. Specifically, cleaned and heat-treated shape memory alloy (SMA) wires were positioned in a custom-designed sample holder in a mold which was subsequently infused with the pre-mixed polymer. The samples were then cured as described in section 2.2. A crucial aspect of the sample preparation was to ensure a precise alignment of the SMA wire to ensure that it entered the polymer matrix at precisely 90°. With that proper alignment of the sample in the loading direction, minimum transverse stresses at the interface will be achieved. The embedded length of the pull-out sample was 50 mm and the free SMA wire length was 40 mm. The embedded part of the specimen had a cross-section of 18 mm x 9 mm.

3. METHODS

3.1 µCT IMAGING

The analysis of the SMA wire surface morphology was performed, using absorption contrast synchrotron radiation-based micro computed tomographic (µCT). To this end, the high energy materials science beamline P07 at PETRA III at Deutsches-Elektronen-Synchrotron (DESY, Hamburg, Germany) was used, which is operated by Helmholtz-Zentrum Hereon. The imaging parameters were: photon energy of 41 keV, with 2501 projections acquired with over 180°, using an exposure time of 100 ms. A Ximea CB500MG detector (Münster, Germany) was used. The (unbinned) effective pixel size was 0.46 µm. Quantitative analysis of the SMA wire surface was performed as described in [4]

The high-resolution 3D imaging enabled, a detailed quantitative description of the applied structured SMA wire surface and the increase of the SMA wire surface due to the structuring of the SMA wire surface could be calculated. The analysis of the µCT data was done as described in [4], with that a threshold value of 4 µm was set.

3.2 SEM IMAGING

The initial characterization of the SMA wire surface after the selective electrochemical etching process was performed using a ZEISS ULTRA PLUS scanning electron microscope (SEM).

3.3 PULL-OUT TEST WITH STRESS OPTICS

The pull-out test was conducted in accordance with the methodology described in [4]. Five specimens were tested for each configuration. Specifically, the embedded section of the sample was secured using a custom-built load frame, the load was applied to the free end, as depicted in Figure 2 (left). The pull-out test speed was set to 25 %/min. The experiment was monitored using stress-optics, as illustrated schematically in Figure 2 (right), which enabled the moment of first interfacial failure to be determined. The analysis of the measured data was done as described in [4].

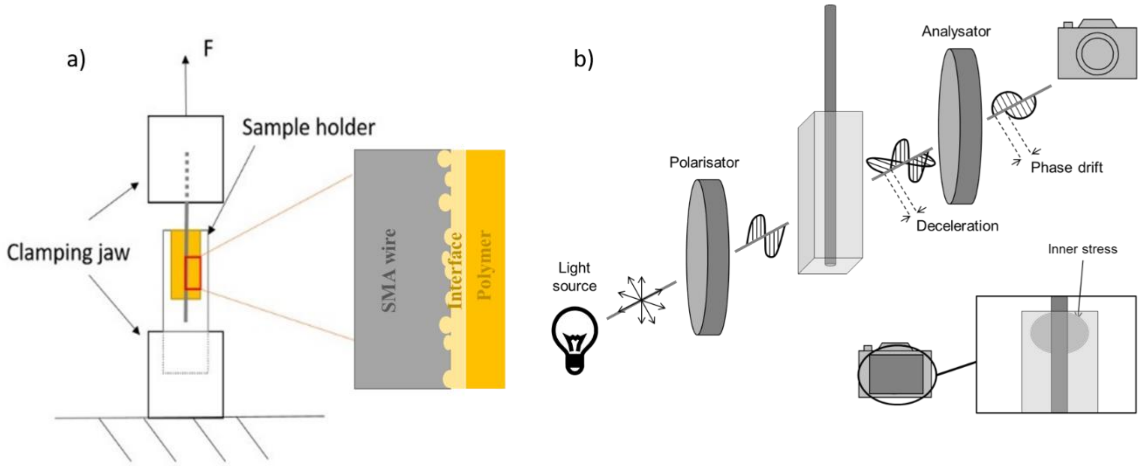

Figure 2: Pull-out test set up explaining mechanical interlocking expected between structured SMA wire and epoxy (a), schematic description of the stress-optic used (b) [13]

4. RESULTS & DISCUSSION

Our results demonstrate that the findings reported in [4] can be extrapolated to SMA wires with smaller diameters (0.5 mm). Surface structuring by selective electrochemical etching can also be performed on 0.5 mm diameter SMA wires in a controlled manner. Figure 3 illustrates the force at first interface failure versus the increase in surface area as measured by µCT for the two structured SMA wire configurations and the untreated as-delivered SMA wire tested in this research. The graph shows the mean values of five samples for each configuration with a 99% confidence interval. As can be seen, both structured SMA wire configurations achieve an increase in the force of the first interfacial failure. The mean values of the first failure force for Structure 1 are 42.6N ± 2.8 N, compared with the as-delivered configuration, an increase of 49.5 % of the first failure force can be achieved at an increase of surface area by a factor of 1.24. This indicates that the improvement in force transmission between the SMA wire and the polymer matrix results not only from the larger contact area but also from the mechanical interlocking. With the Structure 2 configuration, a 14.1% improvement in force transfer, when compared to the as-delivered configuration, is achieved. The force of first failure was 32.2 N ± 1.6 N, and the increase in surface area was a factor of 1.16. The force of first failure of the Structure 2 configuration is significantly lower than that achieved with the Structure 1 configuration. However, the demonstration that the increase in the force of the first failure is not linearly related to the increase in the surface area of the SMA wire is another indication that not only the contact area is important for force transmission, but also the mechanical interlock plays an important role.

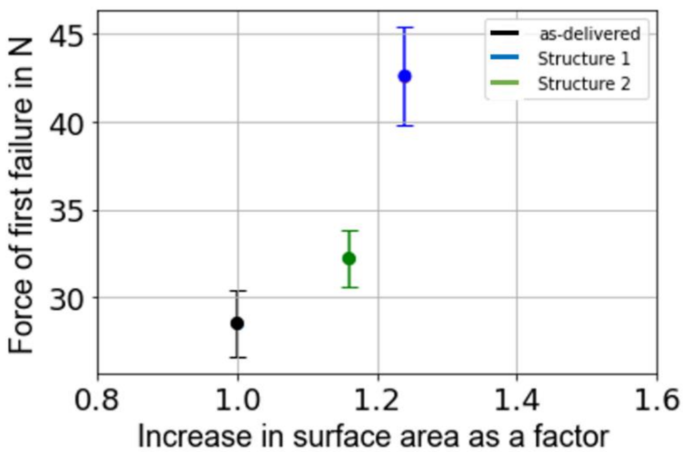

Figure 3: First interfacial force of first failure plotted against the increase in surface areas as a factor for the three different pull-out specimen configurations

To better understand this effect, a comparison of the surface morphology, as characterized by µCT studies, is useful. Figure 4 shows an analysis of the SMA wire surface morphology resulting from selective electrochemical etching for surface structuring. µCT volume data were used for the quantitative statistical description. The analysis of one representative SMA surface is shown for each structured SMA wire surface configuration. The quantitative evaluation of the selective electrochemical structuring by means of µCT analysis indicates distinctly that the distance distribution between the etch pits is significantly larger for Structure 2. Structure 2 has significantly less pits along the measuring length. With a threshold value of 4 µm, 672 pits are found for an evaluated length of 1.5 mm. For Structure 1 there are 1212 for an evaluated length of 1.3 mm. The pits of structure 2 are deeper and larger. The surface morphology parameter "ten nearest neighbour distance" shows the largest quantitative difference when comparing the two structured SMA wire configurations. Although Structure 2 displays deeper pits, Structure 1, which has a greater number of smaller pits, is more efficient in load transmission. Additionally, the geometry of pits can significantly impact mechanical interlocking. While the etch pits in Structure 2 have a simple bowl shape, the etch pits in Structure 1 are hierarchical, with smaller pits nested inside larger ones, creating a more complex path for interlocking with the polymer The SMA wire surface morphology has a large impact on the force transmission and further analysis is needed.

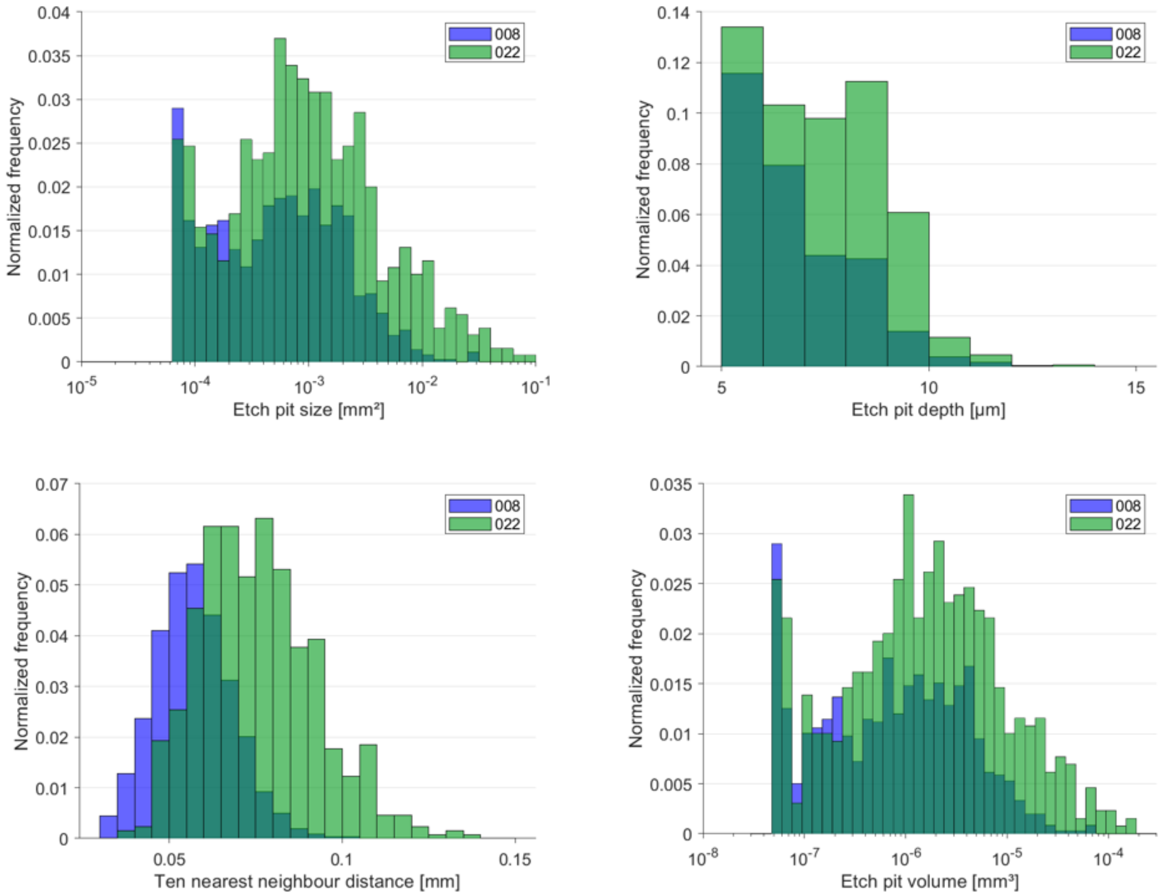

Figure 4: Comparison of two representative specimens of Structure 1 & Structure 2 SMA wire configurations with quantitative statistical analysis of CT data. A quantitative analysis of the surface morphology of the structured SMA wire configurations regarding etch pit size, etch pit depth, ten nearest neighbour distance and etch pit volume is shown.

5. CONCLUSIONS

Using stress optics, the moment of first interfacial failure during the pull-out test becomes measurable. The results show that the force of first interfacial failure can be improved by structuring the SMA wire surface with selective electrochemical etching. With that, a reproducible, quantifiable SMA wire surface morphology can be applied that results in mechanical interlocking by embedding the SMA wire in a stiff polymeric matrix. A correlation between the surface morphology and the improvement in interfacial strength can be made. The SMA wire configuration, which has a significantly larger distance between the single etch pits, higher etch pit depth and size, fails with a lower force of first failure. Using µCT analysis, a detailed statistical description of the surface of the SMA wire can be made, and this can be correlated with the mechanical results obtained from the pull-out test. Hence, a deeper understanding of the failure-driven processes in the interfaces between SMA wire and polymer matrix can be gained.

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