Comportment of the inner interfaces of metal matrix composite wires during thermal loading

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ABSTRACT. The composite wires were makes by an original continuous production technique. The matrix is aluminium, but the fibre tow consists of a bundle of Al_2O_3, SiC or carbon fibres. The first type of load that appeared the permanent bending in elevated temperature. Creep caused the second type of solicitation, and in the case of the third type the temperature was changing cyclically with static tensile loading. The effect of creep and thermal fatigue resulted from the experiments was characterised with the help of microstructure analysis. The examinations show that the damage due to the thermal process has several subsequent parts that also affect the matrix, the fibre tow and their bounding surface.

We have traced the inner interface by sample fracturing. Consequently, the cross-section of the fibre/matrix interface has become analysable. The EDS-analysis of the fibres have shown that there has been a significant concentration change in several elements, and through diffusion the matrix's components have infiltrated into the crystal-lattice of the fibre. Besides, an interfacial layer has formed too that also has an important effect on the mechanical properties of the composite wire.

KEYWORDS: composite wire, creep, thermal fatigue, diffusion, embrittlement

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1. Introduction

A special continuous production technique to produce metal matrix composite wires (MMC wires) has been developed at Northeastern University [1-3]. In this unique process a molten aluminium bath under high-pressure is used to overcome the interfacial energies associated with a non-wetting ceramic or carbon fibre tow to produce MMC wire. The required infiltration pressure depends on wettability of material, diameter and packing density of fibre, for example in the case of carbon fibre in molten aluminium the pressure for good infiltration efficiency can be as high as 8.25 MPa [4]. The infiltration of fibre tow is very quick in this process; the production rate is up to 6 m/min [2].

Many important advantages exist for this processing over the classic techniques (batch- processing, powder metallurgy, diffusion bonding), because this continuous processing shows higher yields and lower costs. The shorter time in molten metal bath and higher infiltration and cooling rates achieved in the continuous process significantly reduce or eliminate fibre degradation caused by chemical reactions with the molten metal. Increases in strength were qualitatively attributed to changes in the matrix microstructure including decreased grain size and amount of second phase segregation. Grain size reduction was correlated with higher pulling velocities [5].

2. Creep tests with bended and tensile samples

For the experiments there have been used composite wire samples with fibre tows of Al_2O_3 (3M Nextel 610) and SiC (Textron) and matrix materials of alloys of Al 99,99 and 2024 respectively. The constant creep resistance has been obtained by bending. During the process a 120 mm piece of wire was fixed into the grip of an arched cannelure with a radius of 50, 75, 100 and 110 mm.

Due to the difference of the cannelure's radius and the wire's diameter (d=1.4 mm) there are some tensile and pressing stress appearing in the wire that present a great variety in their values. The temperatures were 300 and 400°C and the loading process programmed for 1, 2, 4, 7, 14 and 30 days.

Considering the 3 parameters (strain, time and temperature) we looked for concrete details that would lead to rupture. However, the exact time and close details about the rupture process itself cannot be determined with this procedure. Nevertheless, the 'arched' sample broke quicker and into more parts when the radius was smaller. In all cases the so-called 'roof type rupture' can be observed, like presented here in Fig. 1, but which do not happen at ambient temperatures.

The picture shows the rupture of a wire strengthened with Al_2O_3 fibre, where the 'roof line' is located close to the centre of the curve and it is perpendicular on the bent wire's plane. Also, under the 'roof' and very near to the neutral fibre there is some kind of tearing, that is visible on the SiC fibre reinforced wires too.

For the creep tests the MMC-wire is driven through a furnace and the loading is provided by weights fixed at the end of the wire. In this way there is a constant tensile stress of $\sigma = 70$ MPa, given by the applied weight and the cross-section of the wire. Another important determiner of the creep is the temperature, which in the above-presented experiments was 560 degrees Celsius. The creep curves were presented in our previous paper [6].

A laser distance sensor established the strain. The creep strain in an Al_2O_3 fibre reinforced MMC wire after 625 hours is very little. Distinguishable deformations on the surface have not been formed, but in the matrix around impurities and extrinsic phases some cavities have been produced. According to an EDS-

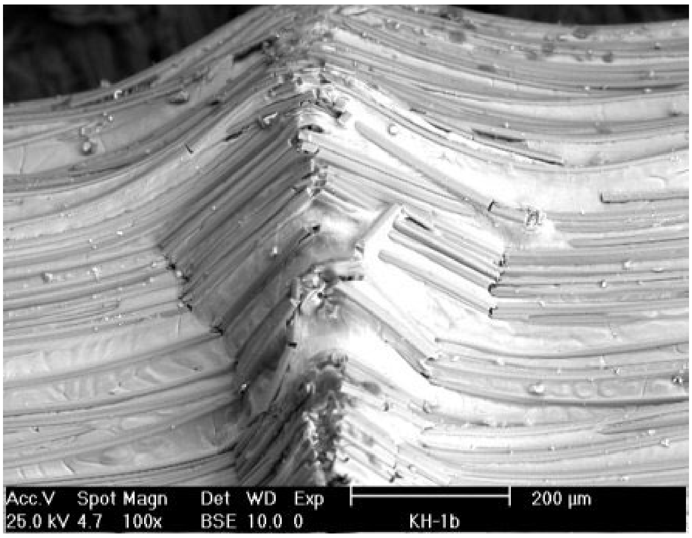

Figure 1. *"Roof type" creep of bended MMC wires*

analysis these cavities contains the following elements: O, Si, S, Cr, Cd, Ti and P.

3. Creep-fatigue tests

The creep-fatigue tests have been analysed in the same way and under similar conditions as presented in part 2. The temperature was fluctuating cyclically between 490 and 540 °C. On the surface of the wires there were visible the so-called ‘solidification waves’. These were caused by the disproportional and discontinuous local or/and directional displacement of the different fibre packs in the finish of processing. Figure 2 shows the wire’s surface at the end of a 1600-hour of testing.

In Figure 2 are shown the oxide layer cracks formed in during the creep. The oxide layer unable to follow the creep strain of the matrix shows cracks too on its surface. At the same time, the fibre itself can also be damaged more or less and due to the locally discontinue strain in some places can become separated from the matrix.

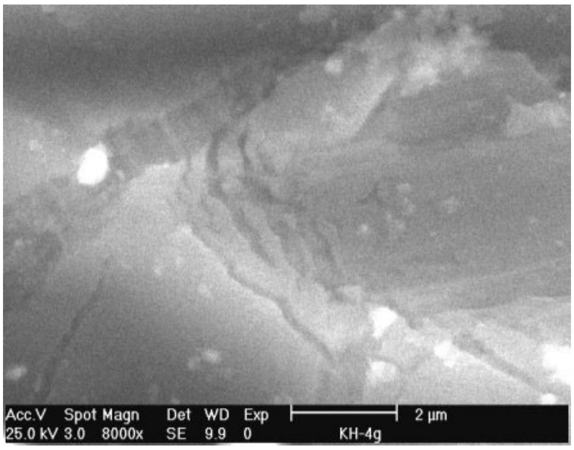

Figure 2. *Oxide layer cracks on the surface of wire*

4. The examination of the inner interface

In our experience, the flexibility of composite wires has changed considerably after the creep test. A wire that is perfect pulling ability in its (basic) production state becomes brittle and practically fracturing at the least bending. It is obvious that this is caused by the inner changes taking place during the stress.

Therefore to trace the inner interface of composite wires, we have used the special but very simple technique of fracturing the samples in controlled atmosphere. This method resembles to the Auger-electron spectroscopy method, however, in this case the sample fracturing was not performed under vacuum conditions but in the chamber of a SEM cleansed with nitrogen. On the cross-section obtained after fracturing are perfectly analysable the characteristic points of the fibre and the matrix and their contact surfaces. In Figure 3 can be seen a portion of the fracture surface. In Figure 3 and 4 it is visible that the SiC fibres sometimes are cleavage lengthways too, and that a mid boundary layer developed between the fibre and the matrix.

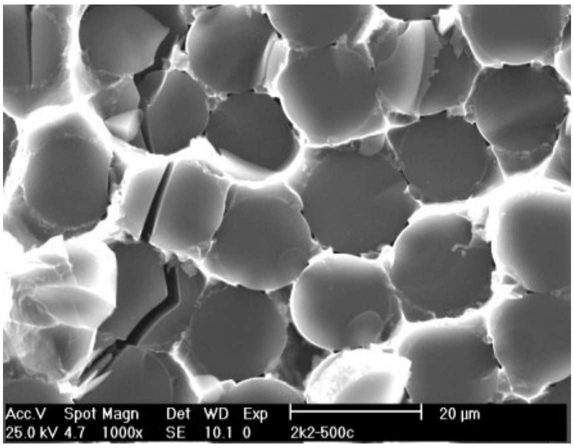

Figure 3. *Fractograph of SiC fibre composite wire*

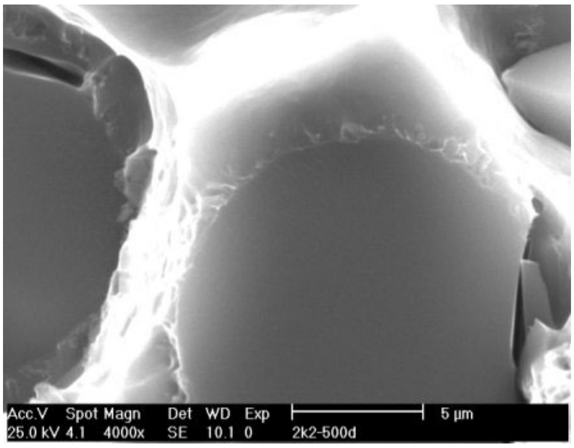

Figure 4. *Boundary layer around the SiC fibre*

	A	B	C	D
C	3,73	30,38	39,24	43,23
O	15,64	6,50	7,83	7,37
Al	57,83	21,76	4,19	1,96
Si	22,67	40,73	47,80	46,38
Ti	0,13	0,63	0,94	1,06

Table 1. Chemical composition measured *in different points of Fig. 5.*

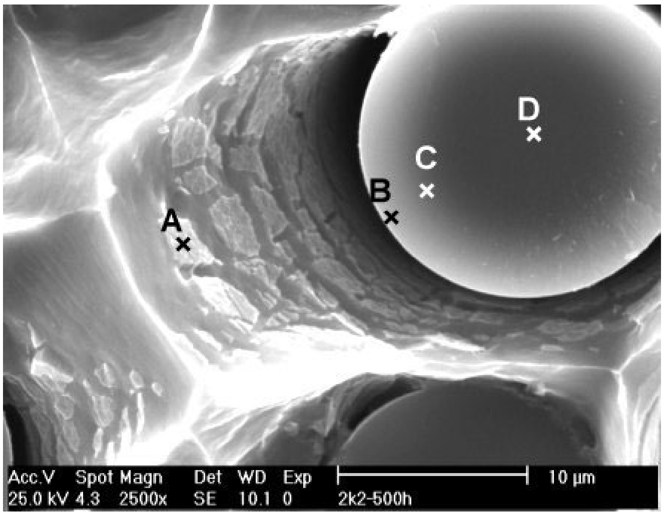

Figure 5. *The compound layer separated from SiC fibre*

Figure 5 shows a fibre that has already lost its boundary layer, which indicates the fact that the layer is better attached to the matrix than to the fibre. Also, the boundary layer shows signs of fragmentation, which means that the layer is brittle. Details about its chemical composition lead to the conclusion that this is a special oxide-silicate type compound layer. Again, there can be observed a range of fracture surfaces where the lateral side of the fibres is also distinguishable and analysable. In such lucky situations even the edges of the fibres are well analysable, and that is due to the fact that the electron beam is not energizing the adjacent matrix. The values measured in characteristic points of the fibre and the compound layer's flakes are all given in chart no.1.Considering the data from the chart, it can be clearly concluded that a diffusion process is taking place in the fibre:

- The Al from the matrix infiltrates into the fibre.
- The Oxygen from the matrix, more precisely from the cavities of the matrix, also infiltrates into the fibre.
- the Ti comes out from fibre and infiltrates into the matrix
- on the compound layers, only a few hundred nm thick, appearing on the fibre-matrix interfaces have.
- An increased content of Al, O, and Si but a low content of C and Ti.

5. Conclusions

Due to the thermal and mechanic loading the composite wires with Al-matrix present a considerable embrittlement, that can be explained with the changes of the reinforced fibre / matrix interface. The thermal activation and the long time intervals produce a considerable diffusion, the process affects the chemical compound of the reinforcing fibre that was homogenous at the beginning. Moreover, on the fibre / matrix interface a compound layer is formed that has a high content of O and Si and as a result its adherence to the fibre is worse than to the matrix.

These two effects are causing embrittlement and deterioration within the bonds of the inner interfaces; therefore the composite wires lose their flexibility in the end. So far we do not possess the necessary experience regarding the tensile stress of the wires and whether is it changing or not. Also, we do not know how much would differ the above-mentioned processes if not a thermal-mechanic loading was applied to the composite. These experiments are still in progress at the moment in our laboratories.

6. Acknowledgement:

The Hungarian Scientific Research Foundation was supported this research: (OTKA T 030004)

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