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Interfacial reactivity in aluminium/carbon fibre composites

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Abstract

The study of the interfacial reactions between carbon fibres and aluminium has been performed using a mass spectrometer. Analysis of the gaseous species evolved by the fibres during heat treatment show that interfacial reactions occur between some of these gases and aluminium. The products of these reactions are aluminium carbide and aluminium oxides. The aluminium carbide produced during heat treatment has been quantitatively measured by determining the amount of methane produced upon the hydrolysis of the carbide.

Key words : carbon fibres, interfacial reactions, metal matrix composites

Introduction

Interfacial reactions in a carbon fibre/metallic matrix composite is a major limiting factor in the development of such materials. Reactions between the fibres and the metal during the manufacture of the composites can lead to matrix embrittlement, fibre degradation and appearance of interfacial brittle phases [1]. These phenomena produce materials of poor mechanical properties and which are susceptible to corrosion [2]. The understanding of the interfacial reactivity is thus essential for the optimisation of fibre/metal pairs and the manufacturing techniques. This paper presents the results of the study of the interfacial reactivity between carbon fibres (M40) and pure aluminium. Analysis of the gases evolved by the fibres during heat treatments showed that reactions between some of the gases and aluminium occur in addition to the reaction between the carbon of the fibres and aluminium.

1. Gas-aluminium interactions

Experimental procedure

Woven carbon fibres were coated with aluminium by physical vapour deposition (PVD) under vacuum. The purity of the aluminium was 99,99% and the thickness of the deposits was 100nm and 200nm. The samples were then heat treated in a furnace

connected to a mass spectrometer in order to follow the gas emissions both qualitatively and quantitatively.

Results and discussion

During the different heat treatments of the carbon fibres, the following gases are evolved : H_2 , H_2O , N_2 , CO , CO_2 . In the presence of aluminium and at temperatures higher than $600^\circ C$, the amounts of H_2O , CO and CO_2 decrease. Figure 1 shows the emission of CO during continuous pumping of the gaseous species evolved from different samples. The more the fibres are covered with aluminium, the less CO is released. In order to verify that the decrease in the emission of CO is due to reactions between aluminium and gases, and not to gases trapped in the aluminium coating, a given pressure of CO was introduced in the reactor and maintained in contact with a woven fibres coated with aluminium (Figure 2). At temperatures higher than $650^\circ C$, the decrease in the pressure of the CO introduced in the reactor points out the reactivity between aluminium and CO . The reactions with CO and CO_2 are assumed to be the following :

As the gases are continuously evacuated out of the reactor, reactions (1) and (2) can happen only at the fibre/metal interface.

However, the main reaction in the carbon/aluminium system is the reaction between the carbon of the fibres and aluminium :

Al_2O_3 produced by reactions (1) and (2) can not be distinguished from Al_2O_3 already present in the aluminium coating which was partially oxidised during the PVD and during the length of time the samples were left in air. Al_4C_3 is produced simultaneously by reactions (1), (2) and (3). The contribution of reactions (1) and (2) to the formation of Al_4C_3 can therefore be estimated by the decrease of CO and CO_2 emissions when aluminium is coated on the fibres. An estimation of the amount of Al_4C_3 formed by reaction (3) can then be made on measuring the total amount of Al_4C_3 formed by all three reactions.

In fact, due to the extremely low amounts of CO and CO_2 released by the fibres during heat treatment, the amount of aluminium carbide produced by the ensuing reactions is much lower than the amount of carbide produced by reaction (3). For example, after a rise in temperature from $500^\circ C$ to $640^\circ C$ at a rate of $5^\circ C/min.$ and after a 30 min. period at a steady temperature of $640^\circ C$, the amount of Al_4C_3 produced by reactions (1) and (2) did not exceed 1,3% of the total amount of Al_4C_3 .

2. Study of the solid species formed during heat treatments

Experimental procedure

Woven carbon fibres coated under vacuum with aluminium were heat treated in order

to form aluminium carbide and put in contact with water vapour at 300°C. In contact with water vapour, aluminium carbide decomposes to release methane. Using mass spectrometry and a volumetric method, the amount of methane was quantitatively detected.

Results and discussion

The decomposition of Al_4C_3 to CH_4 is slow and reaches a maximum after a few hours (Figure 3). Using hydrolysis, CH_4 was detected only at temperatures above 600°C (the samples were held at the specific temperature during 30 minutes). It was found that the amount of aluminium carbide depends not only on the heat treatment, but also on the length of time the samples were exposed to air between the PVD coating and the heat treatment (Figure 4). The decrease in the amount of Al_4C_3 detected with increasing time spans in air can be attributed to the oxidation of aluminium. The oxides formed in air could not react with carbon to form Al_4C_3 .

Knowing exactly the amount of aluminium coated on the woven fibres, it is possible to calculate the percentage of Al_4C_3 produced, assuming that 100% Al_4C_3 would be obtained if all the aluminium had reacted with the fibres to produce carbide. The maximum amount of carbide formed during heat treatments at 700°C and above is 61% after a period of 4 hours in air. The remainder is due to the aluminium oxides formed during the PVD and the period of time the samples were in air. Figure 5 is a profile of the aluminium coating made by XPS on a carbon substrate, showing that metallic aluminium accounts for about 63% of the total deposit.

Conclusion

1. The formation of aluminium carbide at the interface is due not only to the reaction between the carbon of the fibres and aluminium, but also to the reactions between the gases, evolved by the fibres during heat treatment, and aluminium.
2. The study of the hydrolysis of woven carbon fibres coated with aluminium allowed a comparison of the formation of Al_4C_3 for different heat treatments and for different fibres.

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Figure 1 : Thermodesorption of CO for different samples

Figure 2 : Evolution of the pressure of CO introduced

Figure 3 : CH₄ emission

Figure 4 : Dependence of the Al₄C₃ thickness with the length of time the samples were in air for heat treatments of 30min at 700°C (a) and 640°C (b)

Figure 5 : XPS profile of the aluminium coating for metallic aluminium and oxidised aluminium