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REINFORCEMENT CHARACTERIZATION OF METAL MATRIX COMPOSITES BY DSC TECHNIQUE

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The metal matrix composites (MMC) being of very high interest for the aerospace industry, particularly to build up thermal-structural components, it is important to have available methods which are easy and simple to conduct for characterization of the mechanical strength of the material.

This study shows that the differential scanning calorimetry (DSC) may enable us to quantify the energy of cohesion in the fiber/matrix interface area of an aluminium composite reinforced by SiC whiskers.

The energy of cohesion is dependent on the state of reinforcement compared to the matrix strength and take into account both chemical and mechanical characteristics of the cohesion in the fiber/matrix interface area. However, this method would require a systematic calibration based on tests of mechanical characteristics.

Keywords: aerospace industry, DSC, metal matrix composites (MMC)

Introduction

Metal Matrix Composites Materials (MMC), particularly aluminium matrix alloys reinforced with either silicon carbide whiskers or fine particles provide very valuable characteristics: high strength, low density possibility of conventional processing, etc.

Reinforcement of a matrix by fibers, giving consequently higher mechanical properties to the MMC is due to the load transfer between the matrix and the fibers. This transfer being dependent on the quality of the cohesion at the interface, it is very important to characterize the interfacial matrix to stiffener cohesion energy. This characterization allows to evaluate the level of reinforcement of the elaborated MMC material and gives information for optimizing its elaboration.

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This work proposes a thermal analysis technique to evaluate the interfacial stiffener to matrix cohesion energy.

Experimental

Elaboration of the MMC material

Silicon carbide in the form of whiskers economically obtained by a rice hull pyrolysis process is one of the most performant type of reinforcement permitting to achieve very high mechanical characteristics.

The powder metallurgy (P.M.) process begins by mixing the SiC whiskers with alloyed aluminium (1 μm). This blending is performed either through a dry process or using an organic link, it is then cold pressed, hot isostatic pressed between solidus and liquidus phase to produce an aluminium/SiC whiskers billet.

The composite material selected for this study is an aluminium alloy 2024 (aluminium, copper/magmesium) reinforced with 20% SiC whiskers. The billet obtained by P.M. process, has been extruded at 405°C into bars with a diameter of 30 mm, heat treated traditional T6:

– Solution heat treated at 492°C
Temperature elevation during 6 hours
Held at temperature during 6 hours, water quenched
–Waiting time before ageing: 3 days
– Ageing at 190°C
Temperature elevation during 4 hours
Remaining at temperature during 9 hours, air cooling

This traditional T6 heat transfer does not give very high mechanical characteristics to the material because the quench and ageing treatment imposed are not suitable for the following reactions:

i) they get an incomplete dissolution of the precipitate or a beginning of melting in the fiber/matrix interface area during solution heat treating.

ii) they get a perturbation in kinetics of precipitate due to the whiskers which behave as heterogeneous area of nucleation during ageing.

This material will provide a sample of a weak reinforced material. To have a well reinforced material another sample of the same materials has been elaborated with an optimal T6 treatment:

– solution heat treating at 485°C (below the standard T6 which is at 492°C)
– held at temperature of 10 hours (longer than standard T6 which is 6 hours).

This material presents high mechanical characteristics because the longer time at lower temperature permits a better dissolution of the precipitate and avoids the beginning of melting at the fiber/matrix interface. The longer holding time should promote an improvement of the cohesion at the fiber/matrix interface by atomic diffusion in this area. This treatment provides samples of high reinforced material.

Another type of sample has been elaborated from the same matrix material (2024) with 20% carbon fibers produced by pressing alternate layers of matrix and reinforcement, which are then extruded in bars of 20 mm diameter and none adequately heat treated getting a low reinforcement level. This material has been elaborated to serve in a comparative purpose.

Results and discussion

Energetic characterization of the fiber matrix interface

The equipment used is a Perkin Elmer DSC2. The sample weight was between 5 and 20 mg, the heating rate was 20 $deg \cdot min^{-1}$

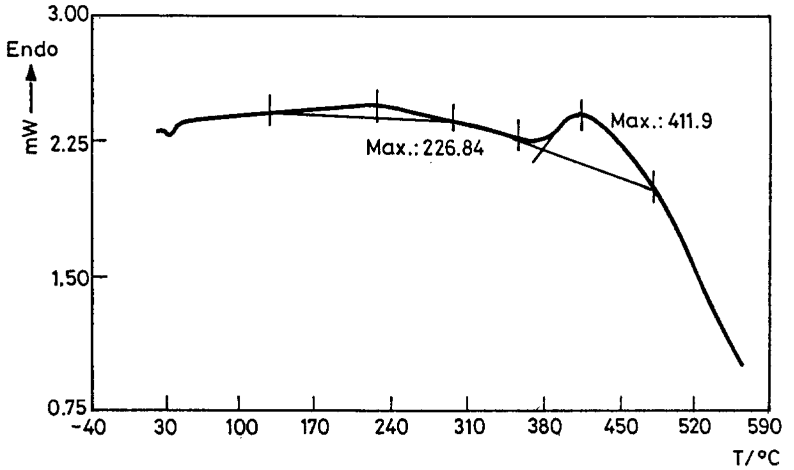


Fig. 1 DSC curve of normalized Al/SiC sample, weight of sample: 12.30 mg, scan rate: 20 $deg \cdot min^{-1}$

DSC curves given by the Al/SiC sample (Fig. 1) show, beyond the usual phenomenon of shift in the solidus due to the matrix, an endothermal peak presenting a maximum about 412°C. This endothermal peak is reproducible with all the other samples (2024/SiC whiskers) produced through the same route. This peak is probably due to a reorganization of the fiber/matrix interface when a well reinforced composite material is heated, because this peak vanishes on the second DSC thermal cycle.

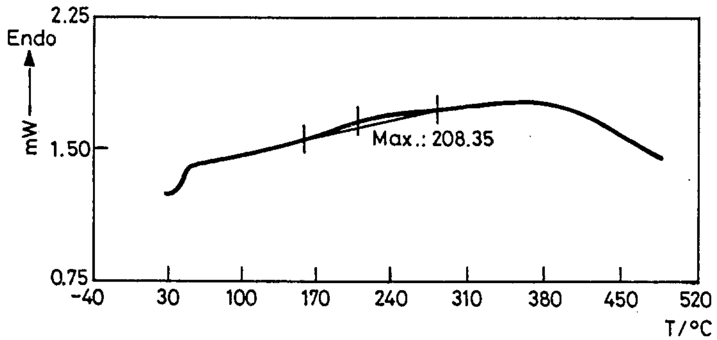


Fig. 2 DSC curve of a low level reinforced Al/C sample, weight of sample: 14.70 mg, scan rate: 20 deg·min^{-1}

This peak does not appear neither for low level reinforced material samples (Fig. 2) nor for the matrix alone (Fig. 3) nor for silicon carbide alone (Fig. 4). This peak does not correspond to a specific transformation of one of those two components.

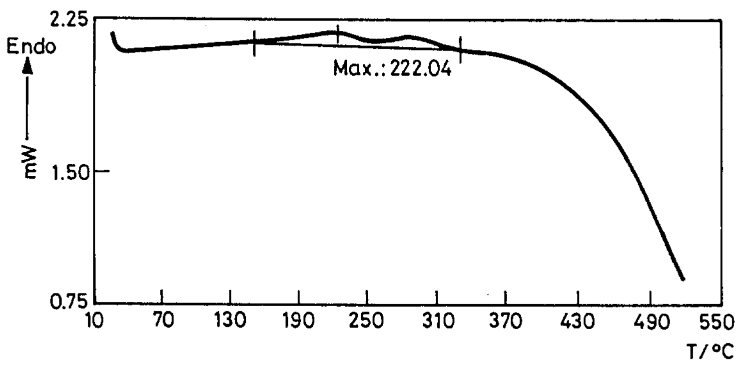


Fig. 3 DSC curve of a reference non reinforced AU 4G sample, (matrix alone), weight of sample: 8.35 mg, scan rate: 20 deg·min^{-1}

A systematic TG realized on the samples (Fig. 5) does not show any mass loss in the temperature levels taken into account.

The reorganization in the material during the thermal cycle is probably accompanied by the liberation of internal compression contraints between the fibers and the matrix. This hypothesis was confirmed by X-ray diffraction studies which showed a level of contraint of 150 MPa on the reinforced sample corresponding to Fig. 1 and no contraint on the low level reinforced ones or not reinforced ones.

Tensile strength trials performed on test parts of the material with high reinforcement level, annealed and examined by electron scanning microscopy show sections with rather smooth SiC whiskers with no tearing off the 2024 matrix, while the not annealed sample observations show the contrary.

These observations confirm the assumption of a link between the type of the heat treatment performed, the compression stresses and the cohesion energy in the fiber/matrix area.

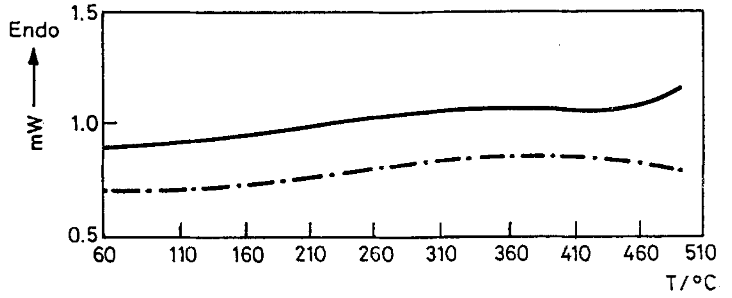


Fig. 4 DSC curve of reference silicon carbide sample, weight of sample: 34.20 mg, scan rate: 20 deg·min^{-1}

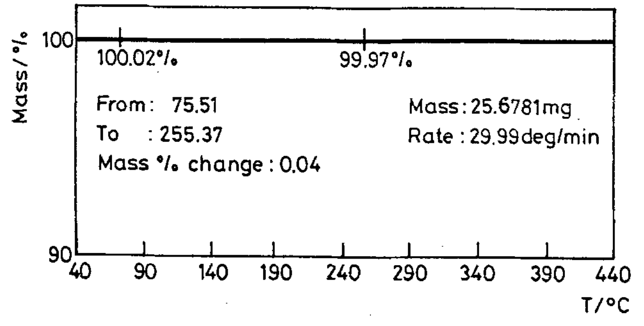


Fig. 5 TG curve of an Al/SiC sample, weight of sample: 25.67 mg, heating rate: 20 deg·min^{-1}

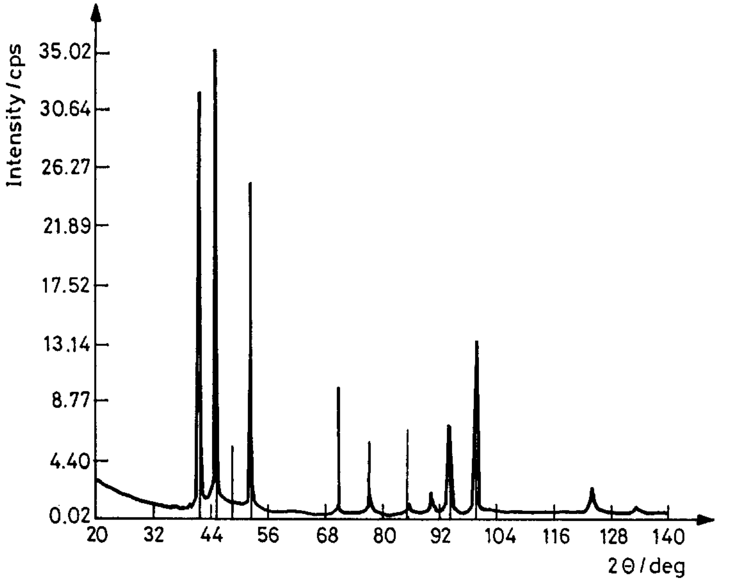


Fig. 6 X-ray diffraction diagram of an Al/SiC sample

Conclusion

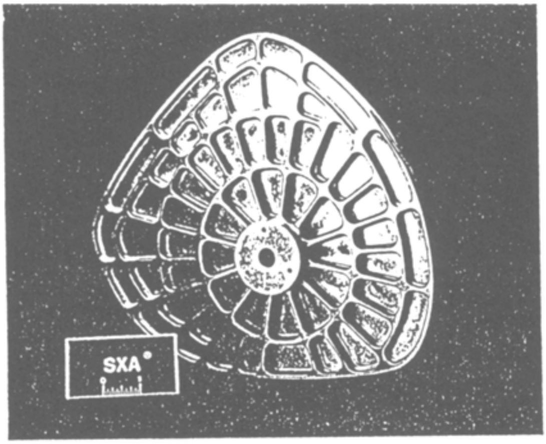
The DSC technique permits to conduct an energetic characterization of the cohesion at the fiber matrix interface by evaluating the organization state of the material at the interface when heating during DSC. This technique allows to better know the reinforcement of a Metal Matrix Composite.

An advantage of this analysis, is to be almost non destructive (a few mg of sample) compared to a conventional mechanical characterization.

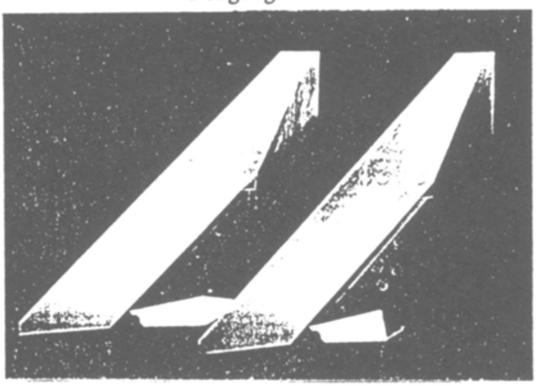
The amplitude of the endothermic peak seems specific of a well reinforced MMC. However, it will be necessary to relate more accurately the percentage of reinforcement to the corresponding endothermic peak by conducting systematic trials on well selected types of materials.

Characterization of the cohesion at the interface by high frequency ultrasonic tests should be performed to make an interesting comparison with the DSC.

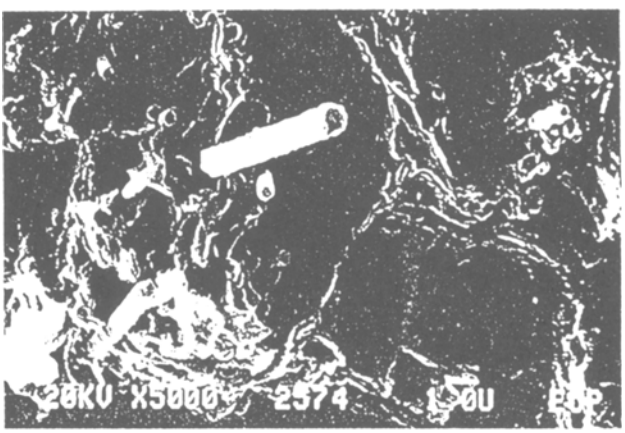
The intensity of the cohesion strength in the interface area is linked to the moistening of the fibers in the matrix and to the compression stress exercised by the metal on the reinforcement which corresponds to thermal-mechanical transformations of the composite (compression extrusion, heat treatment).



Forging



Machining from PM



Microstructure

Fig. 7 Example of components realized in aluminium matrix alloys reinforced with silicon carbide whiskers

Therefore, it would be possible to improve the fabrication process of this type of material (MMC) by performing systematically a DSC at each stage of production.

This would require a calibration of this thermal method (DSC) by correlative trials of mechanical characterization.

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Zusammenfassung — Metallmatrixverbundwerkstoffe (MMC) sind für die Raumfahrtindustrie von großem Interesse, insbesondere zum Bau thermisch-struktureller Komponenten ist es wichtig, Methoden zur Verfügung zu haben, die leicht und einfach zur Charakterisierung der mechanischen Festigkeit der Materialien benutzt werden können.
Es wurde gezeigt, daß DSC es ermöglicht, die Kohäsionsenergie im Fiber/Matrix-Grenzflächengebiet eines mit SiC-Whiskern verstärkten Aluminiumverbundstoffes quantitativ zu bestimmen.
Diese Kohäsionsenergie ist abgängig vom Verstärkungsgrad bezogen auf die Matrixfestigkeit und berücksichtigt sowohl die chemischen als auch mechanischen Eigenschaften der Kohäsion im Fiber/Matrix-Grenzflächengebiet.
In jedem Falle bedarf diese Methode einer systematischen Kalibrierung anhand von Test der mechanischen Eigenschaften.