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Reinforcement Volume Fraction Determination in Metal Matrix Composites

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Abstract: The precise determination of reinforcement volume fraction in metal matrix composites (MMC) is a requirement to control the material quality. Depending on the fabrication technique and materials used, this volume fraction determination poses several difficulties. The metallographic technique, besides being time consuming, is prone to preparation artefacts due to intrinsic phase's hardness that produces polishing relief. The proposed method is based on reinforcement extraction by matrix chemical dissolution. The main requisite is that the chemical solution does not dissolve or attack the reinforcement. A special procedure was established for MMC's where the matrix was an aluminium alloy reinforced with silicon and niobium carbides. The method was standardised by means of special samples prepared by powder metallurgy. The data was checked against metallographic samples evaluated by an image analyser. The method was proven reliable and precise.

Introduction

The evaluation of reinforcement volume fraction in metal matrix composites (MMC) is a requirement that allows the quality control of material production. Depending on the processing route and materials system (matrix and reinforcement), this determination poses several difficulties. The metallographic technique is prone to preparation artefacts due to intrinsic phase's hardness, which produces polishing relief. This technique is also strongly influenced by the ability of the analyst and, if the reinforcement have a large particle size distribution, by the magnification used in the microscope and by the quantitative stereology measurement method.

The proposed method is based on reinforcement extraction by matrix chemical dissolution. The main prerequisite of this method is that the chemical solution does not dissolve the reinforcement. The method is very simple, requiring only basic facilities of a conventional metallographic laboratory.

The method was applied to aluminium alloys reinforced with particulated silicon carbide. Results obtained from standard samples were compared to metallographic data taken from MMC's produced by powder metallurgy. The method was also applied to determine the homogeneity of the MMC's based on the system Al/NbC, in different regions of rods produced via powder metallurgy followed by hot extrusion.

Experimental

Chemical dissolution

In order to isolate the matrix from the reinforcement, it is necessary to find a suitable chemical solution, which dissolves the matrix without affecting the reinforcement. Once the matrix is dissolved, it is possible to separate the intact phase by filtration or decantation. This phase is then dried, weighed and related to the total mass of the sample, to calculate the weight fraction, and the volume fraction. For aluminium or aluminium alloy matrix reinforced with carbides (SiC and NbC), hydrochloric acid is indicated[1] to dissolve only the matrix.

For testing the methodology, samples of known carbide volume fraction were prepared. The materials used were an aluminium alloy AA 2124 and SiC powders. The aluminium and the carbide powder were mixed in an orbital mixer and then, the pellets were compacted. These samples were not extruded. The pellets were dissolved in a 0.1 M HCl solution. The insoluble part was decanted and the remaining solution was extracted using an automated pipette. The reinforcement was successively washed with distilled water. The residue was dried in an oven for 2 hours at 110 □C and then weighed. The results are shown in Table 1.

Table 1. Weight data for a standard sample of aluminium alloy AA 2124 and SiC mixed powders and the recovered residue.

$W_{initial}$ SiC (g)	0.101 ± 0.001
$W_{initial}$ Al (g)	0.428 ± 0.001
W_{total} (g)	0.529 ± 0.002
% $W_{initial}$	19.1 ± 0.3 %
W_{final} SiC (g)	0.102 ± 0.001
% W_{final}	19.3 ± 0.3 %

It can be seen from Table 1 that the weight of the recovered reinforcement is higher than the initial weight. Chemical analysis of the residue showed the presence of aluminium oxide. This is probably due to oxide previously present in the aluminium powder surface. The weight fraction after dissolution (19.3 %) is very close to the initial weight fraction. This shows that the proposed method can evaluate the weight fraction within an error of 1 %.

Metallography

Samples of AA 2124 composite produced by co-deposition of the reinforcement during spray forming were prepared for metallographic analysis and for chemical dissolution. This material was supplied as extruded 20 mm diameter bars. For metallography, samples in the transverse and longitudinal sections were prepared by standard polishing technique with final polishing in colloidal silica (0.25 μm). Fig. 1 shows an optical micrograph of the composite transverse section.

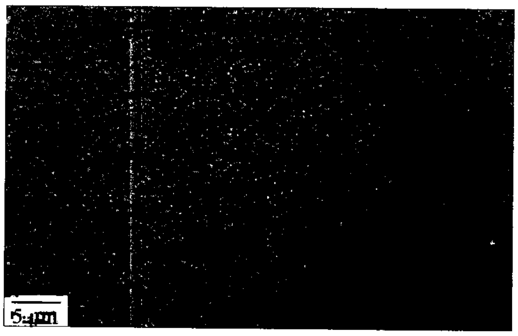

Fig. 1. Cross section optical micrograph of the composite material produced by co-deposition of the reinforcement during spray forming, showing the SiC reinforcement (grey phase) and the aluminium matrix.

The metallographic analysis was performed via image analysis. Fourteen regions of each sample direction were analysed according to ASTM E1245-95 [2]. This material showed different reinforcement area fractions according to the observed section. This was caused by the bar extrusion that produced some reinforcement alignment, see Table 2. Despite of this small inhomogeneity, this composite was used for reinforcement volume fraction determination.

Table 2. Determination of reinforcement area fraction by image analysis for AA 2124 composite and respective reinforcement particle size range.

direction	SiC area (%)	size range (μm)
transverse	21.1	0.2 ± 0.2
longitudinal	23.4	0.1 ± 0.3

Samples from this composite were analysed by the chemical dissolution method and the results showed a reinforcement volume fraction of 20.3 ± 0.3. The data dispersion for the chemical dissolution technique was smaller than that produced by image analysis. However, both methods showed similar results, according to the respective experimental errors.

The chemical dissolution measurement is related to the dissolved macroscopic volume. Hence, it gives the composite average volume fraction. As opposed, image analysis results are related to small regions giving results of a higher dispersion even though the mean value are similar in both cases.

Application

After checking against standard samples and compared its results to image analysis results, the chemical dissolution method was applied as a quality control test. The homogeneity of an aluminium composite produced by powder metallurgy was verified. The composite was a commercial aluminium alloy reinforced with 10 % silicon carbide volume fraction. The composite production route was described elsewhere[3]. From a four meter long extruded bar, three samples were sectioned from three different positions and chemically dissolved. The results of the chemical dissolution method are shown in Table 3. The results show that bars produced by hot extrusion of powders mixture show good homogeneity in their length.

Table 3. Chemical dissolution results of selected sections of an extruded aluminium composite bar.

bar position	reinforcement volume fraction %
begin	9.8 ± 0.2
half middle	9.9 ± 0.2
end	10.0 ± 0.2

Conclusions

The chemical dissolution method proved to be adequate to evaluate reinforcement volume fraction of aluminium alloys reinforced with carbides SiC and NbC. It showed good reproducibility and it is easier to use than the image analysis method.

It is useful method to evaluate the composite on the macroscopic point of view and to check the homogeneity in different regions of a part or element. However, it is not a substitute for image analysis of microscopic regions.

The method was confirmed as a useful quality control test, when applied to extruded Al/NbC composite bars.

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