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Project with NASA entitled

"Advancement of Solidification Processing Technology Through Real Time X-Ray Transmission Microscopy: Sample Preparation"

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Two types of samples were prepared for the real time X-ray transmission microscopy (XTM) characterization. The samples differed in their matrix material being pure aluminum in the first type and an aluminum nickel alloy of eutectic composition for the second type. The added ceramic phase consisted of spherical zirconia particles of 500 µm diameter. The samples were essentially characterized for the three-dimensional distribution of the zirconia particles in the matrix. The above XTM characterization was done for two basic series of experiments.

In the first series directional solidification experiments were carried out to evaluate the critical velocity of engulfment of zirconia particles in the Al and Al-Ni eutectic matrix under ground (1-g) conditions. The particle distribution in the samples was recorded on video before and after the samples were directionally solidified. Knowledge of the particle location prior to the experiment of the cast samples is essential for the evaluation whether the particles were pushed or engulfed during directional solidification, since it is possible to have an area denuded of particles to start with.

In the second series samples of the above two type of composites were prepared for directional solidification runs to be carried out on the Advanced Gradient Heating Facility (AGHF) aboard the space shuttle during the LMS mission in June 1996. The samples in this case were characterized with respect to the zirconia particle distribution before sending them to CNES/France for integration in the cartridge which will be processed in the AGHF.

Experimental:

The following raw materials were used to fabricate the samples:

1.	High purity Aluminum (purity> 99.999%)
2.	Nickel spheres (purity 99.95+%)
3.	Zirconia particles (yttria stabilized ZrO_2 500μm dia.)
4.	Electroless Nickel coating solution for coating zirconia particles.

The composite samples were prepared by the vortex method. A resistance heating pit furnace was used to melt the matrix alloy. Set-up for the mixing of particles in the alloy is shown in Fig. 1. The melts were degassed with high purity Argon gas. Zirconia particles were added to the vortex in the melt created by stirring it with a graphite stirrer. The whole process was carried out under the cover of high purity Argon gas. Approximately 3 vol.% of 500μm dia. zirconia particles were added to the melt. In the case of Al-Ni alloys thethe required amount of Nickel was added to the molten aluminum and the usual degassing and particle addition procedure was followed. The composite was cast in graphite molds. The sample dimensions for ground testing were 9 mm diameter and 100 mm length and for the μ-gravity experiments 10 mm and 168 mm respectively. In overall more than 70 samples were cast. The samples to be used in the μ-g experiment had a thermocouple sheath cast in the middle to

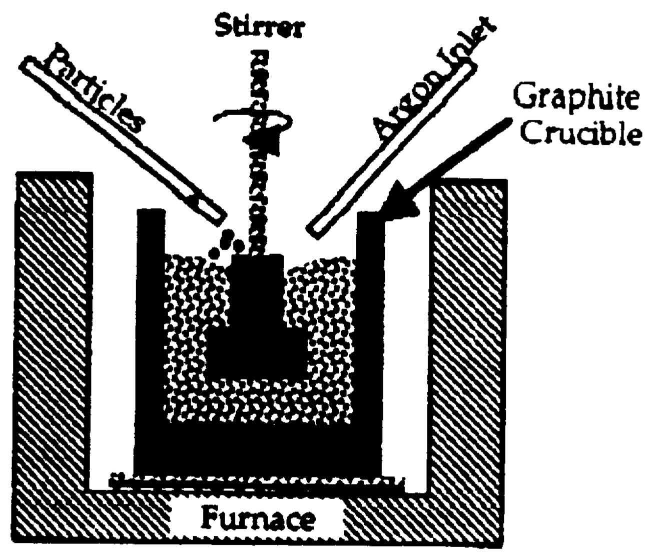

Fig.1 Schematic of the apparatus use to incorporate the particles into the melt by the vortex method

accommodate thermo-couples for recording the temperature inside the sample during directional solidification.

After casting the samples were sent to MSFC at Huntsville for the first stage of sample evaluation. This included the evaluation of the particle distribution throughout the sample, the detection of porosity, the integrity of the cast-in thermocouple sheath, and the verification of the correct positioning of the ceramic sheath. Rotating the samples under the X-ray beam, a three dimensional image of the sample was obtained. Suitable samples were machined to the specifications and again checked under the X-ray microscope for verification of the integrity of the TC sheath. The description of the x-ray microscope is available in the literature. The specimens were examined at magnifications varying from 4X to 20X. Through the CCD camera the X-ray images were recorded on video tapes. Hard copies of the video images could be obtained by importing the image to a Sony color video printer.

Results and Discussion:

Shown in Fig. 2 are two typical pictures of the specimens on which the evaluation was based on. In the picture obtained by X-ray microscopy the zirconia particles (dark spheres) are clearly visible in contrast to the Aluminum matrix, Fig. 2a. For ground experiments the X-ray pictures were converted to schematics where the particle distribution pre- and post-directional solidification are mapped, Fig. 2b. This direct comparison clearly showed the difference in the particle locations before and after the directional solidification run. The speed of directional solidification was in the μm/sec range.

Fig. 2a Example of a sample showing the particle distribution prior to processing using the X-ray microscope.
(Note: the pictures do not represent the same MMC sample)

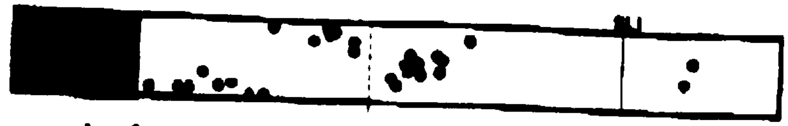

Fig. 2b Example for the evaluation procedure used in this study. The particles map contains the gray unmolten part of the sample, the location of the quench interface (SLI), and the furnace translation distance. (Average Interface Velocity 5 μm/s)

The real challenge was to select the samples for the shuttle experiments. The samples to be selected had to meet the following requirements:

1. The sample should only contain distinct particles, no agglomerates.

2. The particles should be uniformly distributed in the sample.

3. It is very important to have 5-10 particles in the regions to be directionally solidified during the shuttle experiments.

4. There should be no defects of any kind in the regions of interest in the samples meant for the shuttle experiments. X-ray microscopy could detect gas porosity easily.

5. The position and integrity of the thermocouple sheath cast in the specimen should be as desired.

For obtaining a number of eight useful samples for the shuttle experiments and the corresponding ground qualification test about sixty samples were fabricated and tested. In some cases, apart from unsuitable particle distribution, cracking of the thermocouple sheath and in rare instances its bending due to unknown reasons could be detected. This helped in selecting high quality samples for the shuttle experiments.

Science

In a separate study the XTM technique was used to observe the behavior of a zirconia particle at the solid/liquid interface in situ. Up to this experiment the in-situ observation of particle /interface interaction was limited to transparent organic materials. Now one aspect of the proposed theory of particle behavior at the interface could be proven by XTM photographs, Fig. 3. This is a very important contribution regarding the science part of the PEP-si experiment.

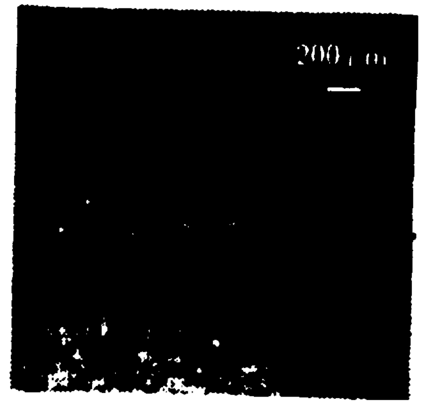

Fig. 3 XTM picture showing trough formation in the bump. Matrix: pure aluminum, Particle: zirconia (720 μm dia.). Interface velocity: 4 μm/s.

Concluding Remarks:

X-ray microscopy proved to be an invaluable tool for characterizing the particle distribution in the metal matrix samples. This kind of analysis helped in determining accurately the critical velocity of engulfment of ceramic particles by the melt interface in the opaque metal matrix composites. The quality of the cast samples with respect to porosity and instrumented thermocouple sheath breakage or shift could be easily viewed and thus helped in selecting samples for the space shuttle experiments. Summarizing the merits of this technique it can be stated that this technique enabled the use of *cast* metal matrix composite samples since the particle location was known *prior* to the experiment.