

## Investigations on the mechanical behavior of 3D interconnected composites developed by liquid metal dealloying

Two challenging ideas were proposed to improve the mechanical properties of 3D interconnected composites produced by liquid metal dealloying. The two approaches were based on the enhanced interface bonding via elaborate alloying design and severe plastic deformation. Firstly, an additional Ni or Al in the Mg melt can act as “glue” on the immiscible interface between FeCr ligaments and the Mg melt phase. Consequently, the work-hardening induced excellent ductility was achieved as stress-drop and restoration signals in the tensile stress-strain curve. Secondly, the high-pressure torsion process leads to structural diversification into the nanolayered structure, probably with improved interface bonding by atomic shuffling.

Metal matrix composites, MMCs, are a material with at least two constituent phases, one corresponds to a metal matrix as a continuous phase, and the other phase should be a different metal or another material such as a ceramic or organic compound. The later phase provides a better level of physical properties such as high strength, high stiffness, or lighter weight to the composite. Conventionally, MMCs are classified on the basis of reinforcing phases depending on their shapes.

Properties of these MMCs are judged by not only each phase's properties itself, but also by several factors such as shape, fraction, size, uniformity of distribution, interface bonding, etc. However, the property changes, which brought by the additional second phase, are highly influenced by the fraction of reinforcements in general. Therefore, the simple rule-of-mixture equation is often used to estimate the properties of MMCs. This tendency indicates that it is hard to overcome the trade-off phenomenon between the two properties. Even some times, some composite materials show a negative hybrid effect.

The visiting professor (Prof. H.S. Kim) and the host professor (Prof. Kato) suggested the new types of composites, which are based on 3D interconnected structure, to obtain a strong positive hybrid effect. Liquid metal dealloying (LMD) is a newly discovered technique in 2011 by the group of Prof. Kato, and this dealloying method can develop individually 3D interconnected two phases by utilizing a molten metal as a dealloying medium [1-6]. Two phases form a continuously disordered structure, and it leads to a large interface area by constructing an interlocked morphology. The size of the features is from nanometer to a few micron scales, which providing much smaller levels compared to those of conventional MMC materials. The interlocked construction can enhance the strength of composite as well as ductility. The interface acts as an efficient barrier against dislocation motion, and this complex geometric structure provides hydrostatic pressure for the brittle phase during deformation. Thus, the crack propagation can be

prevented.

However, 3D interconnected composite developed by a simple LMD triangle has an immiscible character of the interface. Therefore, the interface bonding should be weak. Without good interface strength, strong positive hybrid effects would not be expected. To overcome this undesirable phenomenon, the visiting professor and the host professor started pioneering investigations with two approaches; 1) elaborate alloying design & 2) atomic shuffling via severe plastic deformation.

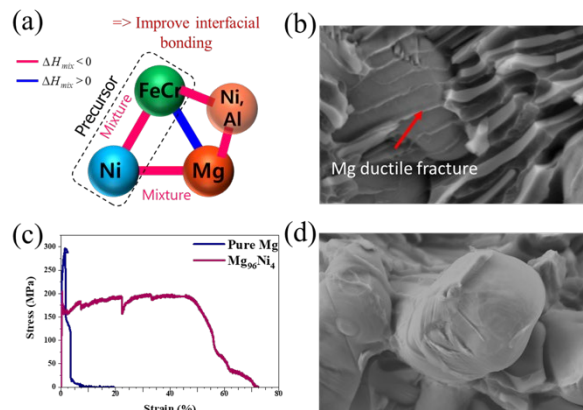


Fig. 1. (a) Modified triangle relationship of the enthalpies of mixing via the elaborated alloying design of the melt. (b) FeCr/Mg composite with additional Ni in the melt. (c) Tensile stress-strain curves of FeCr/Mg composites produced by the LMD process. (d) Fracture surface of the composite with Al.

The first idea is based on carefully designed LMD systems with minor alloying elements in the metallic melt (Fig. 1a). The previous invention considered only a pure metallic melt and the simple miscibility relationships among the precursor elements and the metallic melt element for the selective dissolution of a miscible component. Two different minor alloying elements were selected to study the role of its chemical affinities with precursor elements and the metallic melt element. In the simple LMD

system of  $(\text{Fe}_{80}\text{Cr}_{20})_{50}\text{Ni}_{50}$  precursor and Mg melt, 4 at.% of Ni or Al was added in the Mg melt. The minor alloying element of Ni resulted in the eutectic structure of Mg and  $\text{Mg}_2\text{Ni}$  intermetallic. The high concentration of Ni in the melt also led to the enhanced interface bonding between FeCr ligaments and those melt phases (Fig. 1b).

Mg composite with  $\text{Mg}_2\text{Ni}$  intermetallic has been known as extremely brittle. However, in the complex geometry of the LMD composite, the fracture of  $\text{Mg}_2\text{Ni}$ , as well as Mg, can be delayed. Furthermore, enhanced bonding strength prevents crack propagation through the 3D interlocked morphology. Consequently, the work-hardening induced excellent ductility could be achieved as stress-drop and restoration signals in the stress-strain curve (Fig. 1c).

The minor alloying element of Al also demonstrated the improved interface bonding on the fracture surface. Pure Mg melt sample clearly showed the distinct debonding behavior during the tensile loading, and it exhibited limited elongation. Near the interface, ligaments and melt phase depicted different levels of plastic deformation. On the other hand, most of the interface area was tightly bonded in the Al added sample even after a fracture (Fig. 1d). Al is miscible to both ligament elements (Fe and Cr) and Mg melt. The added 4.0 at.% of Al did not produce the second phase, but it produced a solid solution phase with Mg. Furthermore, the high chemical affinities of Al with Fe and Cr cause the bulk diffusion of Al atoms into the ligament, and they might act as “glue” at the immiscible FeCr-Mg interface.

Severe plastic deformation was the second idea. The high-pressure torsion (HPT) process can apply shear strain over  $\sim 100$  under high hydrostatic pressure without fracture (Fig. 2a). The disordered interlocked composite structure of LMD was diversified into the nanolayered structure (Fig. 2b). Not only the structural changes and grain refinement, but also it would be useful to improve interface strength through atomic shuffling via severe deformation. Thus, the HPT processed LMD composite would result in high strength and good ductility. Furthermore, the limited path along the vertical direction of the interlocked nanolayers has a high potential to develop new nanocomposite materials for diffusion or thermal barriers.

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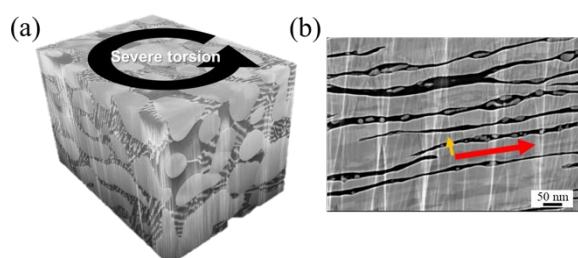


Fig. 2. (a) 3D microstructure of the LMD composite and schematic diagram of shear strain by HPT process. (b) Interlocked nanolayers after the HPT process.

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 Hidemi Kato (Non-Equilibrium Materials Research Laboratory)  
 E-mail: hikato@imr.tohoku.ac.jp  
 URL: <http://www.nem2.imr.tohoku.ac.jp/index-e.html>

Hyoung Seop Kim (Pohang University of Science and Technology)  
 Email: hskim@postech.ac.kr