

## DEVELOPMENT OF MATERIALS PROCESSING USING MAGNETIC FIELD

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### ABSTRACT

**DEVELOPMENT OF MATERIALS PROCESSING USING MAGNETIC FIELD.** The magnetism, the well-known physical phenomena, has been applied for developing the magnetic materials and devices. To improve the performance of the magnetic materials is obviously important issue in engineering until now. Since the cryogen-free superconducting magnets enable us to impose the relatively high magnetic field such as 10T in the room temperature bore with diameter from 50 mm to 300 mm for weeks, different attempts using the magnetism have been proposed for developing materials processing. The responses of the paramagnetic and the diamagnetic materials to the high magnetic field are often detectable. The high magnetic field has potential to control the micro structure of the ferromagnetic, the paramagnetic and the diamagnetic materials. Therefore, electromagnetic processing of materials (EPM) using the high magnetic field has been realized as a unique technique for controlling the microstructure. The high magnetic field was used to achieve the crystallographically aligned structure, to produce the regular structure in which one phase is regularly aligned in the matrix, to develop the containerless processes by the magnetic levitation and so on. In this paper, the principle of EPM is briefly explained. Then, some applications of the high magnetic field to the materials processing are presented; (1) the crystallographically aligned structure of the Bi-Mn system and (2) the regular structure formation in the Al-In monotectic alloys.

**Key words :** Magnetic Field, electromagnetic processing of materials (EPM)

### ABSTRAK

**PENGEMBANGAN PROSES BAHAN MENGGUNAKAN MEDAN MAGNET.** Kemagnetan, sebagai fenomena fisika yang sudah dikenal, telah diaplikasikan untuk mengembangkan bahan dan peralatan magnetik. Peningkatan kinerja bahan magnetik telah menjadi isu penting di bidang rekayasa sampai saat ini. Karena magnet superkonduktor bebas-*cryogen* mampu meningkatkan medan magnet yang relatif tinggi, misalnya 10T pada temperatur ruang dengan diameter dari 50mm sampai 300mm selama berminggu-minggu, beragam usaha dengan memanfaatkan kemagnetan telah diusulkan untuk pengembangan proses bahan. Respon dari bahan-bahan paramagnetik dan diamagnetik terhadap medan magnetik tinggi seringkali dapat dideteksi. Medan magnetik tinggi memiliki potensial untuk mengontrol struktur mikro bahan-bahan ferromagnetik, paramagnetik dan diamagnetik. Oleh karena itu, proses elektromagnetik bahan (*EPM*) dengan menggunakan medan magnetik tinggi telah dilakukan sebagai suatu teknik yang unik untuk mengontrol struktur mikro. Medan magnetik tinggi telah digunakan untuk mendapatkan struktur yang tertata secara kristalografis, guna menghasilkan struktur reguler dimana satu fase tertata secara reguler pada matriks, untuk mengembangkan proses tanpa kontainer dengan levitasi magnetik dan lain-lain. Pada laporan ini, prinsip EPM dijelaskan secara ringkas. Kemudian, beberapa aplikasi medan magnetik tinggi terhadap proses bahan dipresentasikan; (1) struktur yang tertata secara kristalografi dari sistem Bi-Mn dan (2) pembentukan struktur reguler pada paduan monotektik Al-In .

**Kata kunci :** Medan magnet, *electromagnetic processing of materials (EPM)*

### INTRODUCTION

In general, magnetism is one of the well-known physical phenomena in which attractive or repulsive forces are caused between materials. The magnetism has been used for various kinds of magnetic materials and devices. For example, magnets are the materials that produce a magnetic field spontaneously. So many

studies have been extensively performed in order to improve the magnetic properties. Namely, the researches on the magnetic materials have paid much attention to exert the favorable magnetic properties. To improving the performance of the magnetic materials is obviously important issue in engineering until now.

Recently, slightly different attempts to use the magnetism for controlling microstructure of various materials have been made. The magnetic effects for nickel, iron and their alloys that exhibit ferromagnetic properties are easily detectable. However, even paramagnetic and diamagnetic materials have responses to the magnetic fields. The responses of paramagnetic and diamagnetic materials are often negligible under a conventional magnetic field (ca. less than 1T).

The magnetic energy for paramagnetic (diamagnetic) and ferromagnetic materials is expressed by the following equations, respectively.

$$E = -\frac{1}{2}\chi H^2 \text{ (para- and diamagnetic) } \dots\dots (1a)$$

$$E = -MH \text{ (ferromagnetic) } \dots\dots\dots (1b)$$

As shown in the above equations, the magnetic energy can clearly exist if the magnetic field is sufficiently high. Namely, the magnetic properties and the magnetic effect for the paramagnetic and the diamagnetic materials can be detectable under the high magnetic field.

In 1990s, the cryogen-free-type superconducting magnets (Fig.1), which does not need liquid He supply, have been developed by Japanese companies. The magnets enable us to impose the high magnetic field up to 15T in the room-temperature bore (50-300mm in diameter) for the long duration (more than one month). The features of the magnets are very beneficial for materials processing. Thus, the high magnetic field was realized as a tool for controlling the macrostructure of the various materials. Electromagnetic processing of materials (EPM) using the high magnetic field has extensively studied in the last decade [1-4]. This paper briefly reviews EPM using the high magnetic field and the recent results in our group.

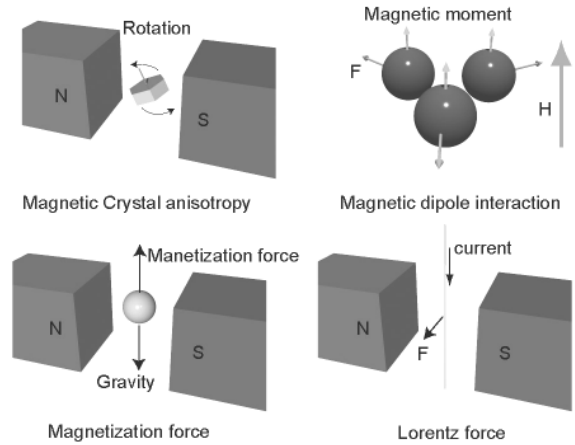


Figure 1. An example of the cryogen-free magnets. Typical intensity of magnetic field is 10T in a bore with diameter of 100mm. (<http://www.jastec.org/>)

## HIGH MAGNETIC FIELD FOR CONTROLLING MICROSTRUCTURE OF MATERIALS

Figure 2 shows the magnetic effects that can be used for controlling microstructure of materials. All of the effects are well known in physics. However, the effect is almost negligible for the paramagnetic and the diamagnetic materials when the conventional magnet is

used. Thus, there were very few researches in which the effects were used to control microstructure. As mentioned in the previous section, the effects can be detectable even for the paramagnetic and the diamagnetic materials under the high magnetic fields.



Several applications for controlling microstructure of materials have been proposed by using the high magnetic field. The microstructures obtained by using the high magnetic field are presented in this section.

### Crystallographically aligned structure

When the particles consisting of the single grain with the magnetic anisotropy suspended in a fluid under a magnetic field, the particles tend to rotate in the favorable direction with respect to the magnetic anisotropy energy. Thus, the materials in which the grains crystallographically align can be fabricated by using the crystal magnetic anisotropy. Details of the rotation mechanism for the crystal alignment under a magnetic field were discussed in ref. [1,2].

For the paramagnetic and the diamagnetic materials with asymmetric unit cells, the magnetic susceptibility depends on the crystal orientation. It should be emphasized that all of the materials such as metals, oxides, inorganic materials, organic materials, polymers, protein and so on can exerts the paramagnetic or the diamagnetic properties. Thus, the crystallographically aligned structure can be obtained for various materials.

For the sake of simplicity, one considers a crystal with a hexagonal unit cell. The magnetic susceptibility of the crystal is expressed by

$$\begin{aligned} \chi(\theta) &= \chi_a \sin^2 \theta + \chi_c \cos^2 \theta \\ &= \chi_c + (\chi_a - \chi_c) \sin^2 \theta \end{aligned} \dots\dots\dots (2)$$

Here  $\theta$  is the angle between the c-axis and the magnetic field. The magnetic energy is given as a function of the angle (Fig.3(a)).

$$E = -\frac{1}{2} \chi(\theta) H^2 \quad \dots\dots\dots (3)$$

If  $\chi_c$  is larger than  $\chi_a$ , the c-axis of the crystal tends to align in the magnetic field (Fig.3(b)). Even if  $\chi_c$  is smaller than  $\chi_a$ , the rotating magnetic field in plane 1-2 achieves the crystallographically aligned structure (Fig.3(b) and (c)) [2].

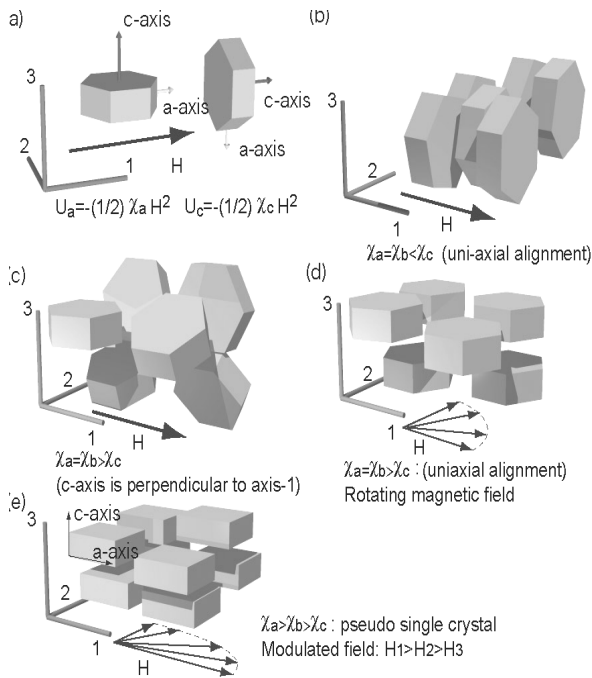


Figure 3. Schematic illustration of the crystallographically aligned structure.

In the case that  $\chi_a$ ,  $\chi_b$  and  $\chi_c$  are different each other, the modulated magnetic field in plane 1-2 can achieve the pseudo single crystal [5].

For the ferromagnetic materials, the magnetic anisotropy exists even in the cubic unit cells. Thus, the easy magnetization direction of the ferromagnetic crystal also tends to align in the magnetic field.

The uni-axial crystal alignment and the tri-axial crystal alignment (pseudo single crystal) can be achieved for various materials by suspending the grains with the magnetic anisotropy under the high magnetic field. In this fabrication route, the green consisting of the crystallographically aligned grains has to be sintered.

Recently, the crystallographically aligned structure could be obtained even in the solid transformation (coarsening, Ostwald ripening) [6-8]. This result showed that the crystal alignment can be obtained by the non-rotation mechanism. The experimental results and the application for the magnetic devices are presented in the following section.

### Regular Structure

As shown in Fig.2, the repulsive force operates between the particles with the same magnetic moment in the plane perpendicular to the magnetic field. The

repulsive force between the particles can contribute to the regular structure in which one phase are regularly aligned in a matrix. In addition to the magnetic dipole interaction, the Lorentz force induced by the high magnetic field contributed to formation of the regular structure during the unidirectional solidification of some metallic alloys [9].

As an application of the high magnetic field for the regular structure formation, fabrication of the porous materials by using the solidification under a magnetic field is also presented in the following section.

### Levitation

Levitation of various materials has some advantages. The liquid phase can be suspended without any contact with the solid crucible wall, the contamination from the crucible can be avoided, and so on. Thus, the development of the levitation technique has been required in various research fields.

The magnetic energy and the potential energy due to the gravity is given by

$$U = \left( \rho g z - \frac{1}{2} \chi H^2 \right) V \quad \dots\dots\dots (4)$$

Here, V is volume of the materials and z is the position in the vertical direction. Thus, the force acting on the materials in the vertical direction is given by

$$F = \left( -\rho g + \chi H \frac{\partial H}{\partial z} \right) V \quad \dots\dots\dots (5)$$

If the magnetization force is sufficiently large, the force F can be zero. Thus, the material can be levitated by the high magnetic field. For the stable levitation, the relative magnetic susceptibility has to be negative. The first experiment of the levitation by using the high magnetic field was demonstrated for water, wood and polymers [10]. The magnetic levitation was also used for the materials processing [11].

For the metallic materials, the levitation technique using the alternating magnetic field has been widely used to measure some thermophysical properties [12] and to examine solidification in the undercooled melt [13]. There is an inevitable disadvantage in the levitation technique. The alternating magnetic field causes the lift force and simultaneously the stirring force. As a result, the oscillation and the intense convection always exist in the levitated melt. The oscillation and the convection degrade the accuracy of the measurement and significantly influence the solidified structure.

Thus, the levitation technique that enables us to levitate the metallic melt without the oscillation and the convection was desired. Recently, the levitation technique using the alternating and the static magnetic field overtake the disadvantage [14].

## CRYSTALLOGRAPHICALLY ALIGNED STRUCTURE OF Bi-Mn SYSTEM

### Alignment During Coarsening Process (Solid State)

It has been recognized that high magnetic fields can be a powerful tool to achieve the crystallographically aligned microstructure even for paramagnetic and diamagnetic materials [15-30]. In most processed for achieving the aligned structure, the crystals with the magnetic anisotropy, which are suspended by a fluid, rotate in a favorite direction with respect to the imposed magnetic field. It is also reported that the transformation in solid state under a magnetic field can produce the crystallographically aligned structures.

The aligned microstructures in Bi-Mn alloys were produced by coarsening process under a magnetic field [23,24]. The BiMn compound exhibits the large crystal magnetic anisotropy energy [25]. The experimental results indicated that the magnetic energy contributed to selection of the coarsening process. This section presents the aligned structure formation of the Bi-Mn alloys during the coarsening under a magnetic field.

Bi-20at%Mn alloys were rapidly solidified. Bulk specimens were made of the rapidly solidified particles by cold press (350MPa). Dimension of the specimens was 6 mm in diameter and approximately 2 mm in thickness. The bulk specimens were sealed in a SiO<sub>2</sub> tube under several 10 torr Ar gas and then annealed under a magnetic field. Strength of the imposed magnetic field ranged from 0T to 10T. Annealing temperature was 513K, which is below the eutectic temperature of Bi and BiMn. Thus, the solid state transformation occurred during annealing. Distribution of c-axis of the BiMn compound was measured by the X-ray diffraction technique.

Figure 4 shows the microstructures of the Bi-20at%Mn alloys. In the rapidly solidified Bi-Mn specimens, the BiMn grains with radius less than 1 μm were uniformly distributed in the Bi matrix. Mn was supersaturated in the Bi matrix due to the rapid solidification. Coarsening of the BiMn grains in the solid Bi matrix occurred during annealing at 513K. As shown in Fig.4, size of the BiMn grains increased with increasing the annealing time.

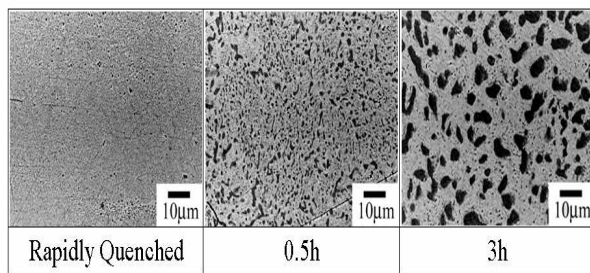


Figure 4. Microstructure of the Bi-20at%Mn alloys. Black: BiMn compound, gray: Bi matrix

Figure 5 shows distribution of c-axis of the BiMn grains annealed at 10T. For the specimen annealed for 0.5h, the crystallographic alignment was not detected in the stereoprojection. In contrast, it was clearly observed that c-axis of the BiMn grains tended to be parallel to the imposed magnetic field. The experimental results clearly indicated that the BiMn grains with the favorite orientation preferably survived during the coarsening under a magnetic field. As a result of the grain selection, the crystallographically aligned structure was obtained during the solid state transformation (coarsening).

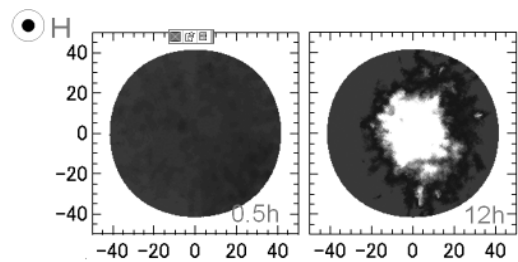


Figure 5. Stereoprojection of the BiMn c-axis during annealing under a magnetic field of 10T.

A model for explanation of the crystallographic alignment during coarsening under a magnetic field has been proposed [31]. Figure 6 shows a schematic illustration of the grain coarsening under a magnetic field. The BiMn phase is randomly distributed in the matrix phase (the arrows indicate the easy magnetization direction). The free energy of the BiMn grains depends on the crystal orientation with respect to the magnetic field. The BiMn grain of which the c-axis is parallel to the magnetic field has the lowest free energy among the BiMn grains. The solute concentration (of the Bi matrix) at the interface between the Bi matrix and the BiMn is

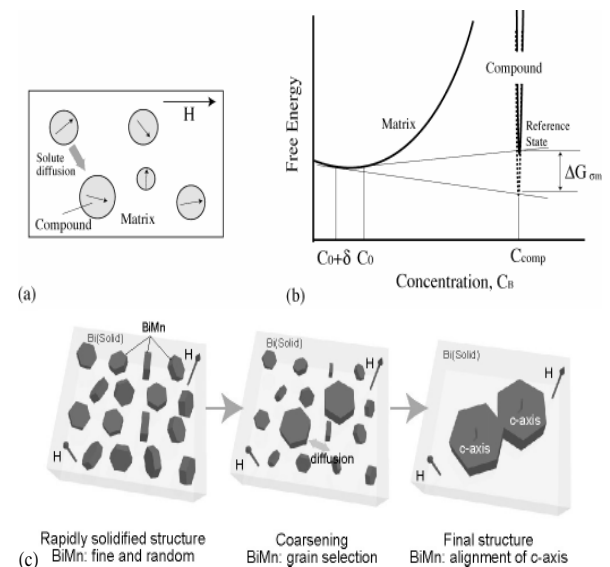


Figure 6. Coarsening of the BiMn grains under a magnetic field. (a) Distribution of the BiMn grains, (b) free energies of the BiMn and the Bi matrix and (c) evolution of the crystallographically aligned structure during annealing.

determined by the grain size and the crystallographic orientation with respect to the magnetic field. As a result, the solute transfer takes place between the grains and consequently promotes growth of some grains with the favorable orientation concurrently with annihilation of some grains.

The results proved that the crystallographically aligned structure can be produced even in the solid state transformation. Namely, the non-rotation mechanism can be used for controlling the crystallographically aligned structure. The effect observed here is called the magneto-thermodynamic effect.

### Crystal Alignment of Bi-Mn by Laser Melting Under a Magnetic Field

The magnetically textured structures of the Bi-Mn alloys were produced by remelting the rapidly solidified specimens [32] and by annealing the rapidly solidified specimens [33] under a magnetic field as mentioned in the previous section. The previous results suggested that the aligned structure could be produced in a desired region if the melting or the coarsening region was controlled by the laser irradiation. It means that the laser melting and annealing can be applied to fabrication of the micro-magnetic devices.

The micro-magnetic devices have been proposed for micro-electromechanical systems (MEMS). For example, mm-size motor was fabricated by using Nd-Fe-B thin film magnet [34-36]. Micro fluid control system was also proposed using the magnetohydrodynamics [37]. Thus, it is of interest to examine the feasibility of the semi-solid solidification and the coarsening in the Bi-Mn system from a viewpoint of micro-magnetic device fabrication.

Figure 7 shows an experimental apparatus for laser irradiation under the magnetic field. The static magnetic field was imposed by a cryogen-free superconducting. The specimen was placed at the maximum position of the magnetic field. Ar gas was inserted into the specimen chamber to avoid oxidation. LD laser (wave length: 834 nm, maximum power: 1W) was used for the irradiation

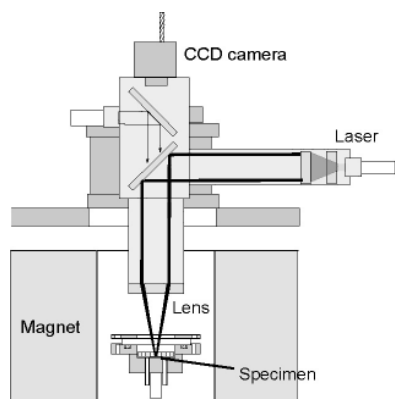


Figure 7. Experimental apparatus for laser irradiation under a magnetic field

through an optical fiber. The irradiated region on the specimen surface was approximately 10 mm in diameter. Compacts of the rapidly solidified Bi-20at%Mn particles were prepared by cold press under a pressure of 350MPa. The leakage magnetic flux at the surface was detected by a magnetic force microscopy (MFM) without an external magnetic field.

Figure 8 shows the images of the optical microscope and the magnetic force microscopy (irradiated region: 10 mm in diameter, laser power: 1W). The Bi-Mn grains with diameters of 1-2 μm were distributed in the selectively solidified or coarsened region. The leakage magnetic flux from the Bi-Mn grains was clearly detected in the selectively solidified region, although the magnetic flux could not be detected in the matrix.

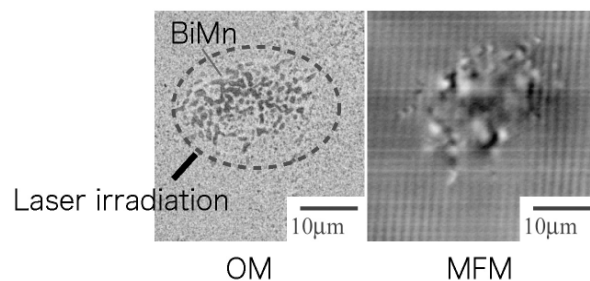


Figure 8. Images of the optical microscope (OM) and the magnetic force microscopy (MFM). OM: the grains with dark gray is the BiMn grains coarsened under 10T. MFM: Bright and dark region indicate the magnetic flux on the surface

The experimental result proves that the semi-solid solidification and the coarsening by the laser melting under a magnetic field can produce the aligned structure of the Bi-Mn in the selected region as small as 10 mm in diameter. In other words, the micro-magnet (Bi-Mn) can be fabricated by the laser annealing. The process will be beneficial for producing the micro-magnetic devices.

### FORMATION OF THE REGULAR STRUCTURE BY SOLIDIFICATION UNDER MAGNETIC FIELD

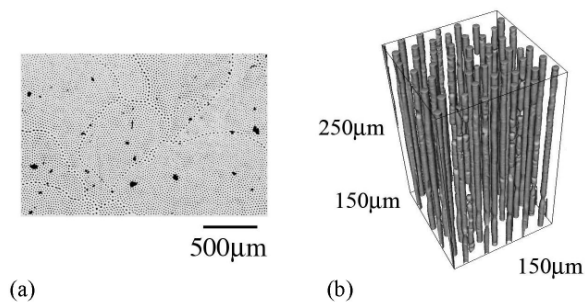
The high magnetic field was used to suppress the microscopic melt flow and motion of immiscible droplets of which the diameter was typically 10 μm. The suppression is called micro-MHD (magnetohydrodynamic) effect. The regular structure in the monotectic alloys were produced by using the micro-MHD effect and the magnetic dipole effect.

A monotectic reaction is defined by the simultaneous production of a solid phase (S) and a liquid phase (L1) from a liquid phase (L),  $L \rightarrow S + L1$ . It is well known that the aligned rod structure can be produced for monotectic alloys in monotectic compositions by unidirectional solidification as well as for eutectic alloys [38-40]. Compared to eutectic structures [41], very

specific growth conditions are required for developing the aligned structure in monotectic alloys. For example, a regular structure has rarely been produced in hypermonotectic compositions.

In spite of the difficulties associated with the monotectic solidification, there are some attractive features of the aligned structure of monotectic alloys. One is that the shape of the minor phases is truly cylindrical because they are liquid while major phases solidify. The other is that minor phase rods of the same diameter are regularly aligned with each other, because the major and minor phases grow cooperatively. It is a sort of self-organization process. If the fibrous minor phase is removed from the matrix, porous media in which deep pores with the same diameter are regularly aligned in the matrix can be fabricated [42,43]. Thus, it is of interest to control the monotectic solidification.

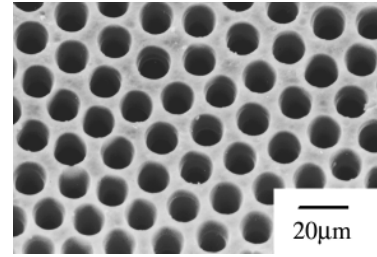
Hypermonotectic Al-10at%In alloys have been unidirectionally solidified under a magnetic field of 10T [43]. As shown in Fig.9, the imposition of a static magnetic field during unidirectional solidification successfully achieved an aligned rod-like structure, even for the hypermonotectic composition (10at%In). The 3D image was reconstructed from computerized tomography using a synchrotron radiation facility [44]. The continuous In rods with diameters of 10 - 20  $\mu\text{m}$  are regularly aligned parallel to each other.



**Figure 9.** (a) Transverse section of Al-10at%In monotectic alloys solidified at 10T and (b) 3D image obtained by micro X-ray CT. The black and gray are Al and In, respectively. In the 3D image, the Al phase was removed.

Electrochemical dissolution successfully removed the In rods from the matrix [43,44]. Figure 10 shows the porous Al produced using the monotectic alloys solidified under the high magnetic field. Deep pores whose depths were more than 500  $\mu\text{m}$  were produced by monotectic solidification under magnetic field and electrochemical dissolution.

The study implies that a high magnetic field is a powerful tool for controlling the microstructure and contributes to the fabrication of functional materials. The micro-MHD effect and other magnetic effect will integrate the high magnetic fields into materials processing.



**Figure 10.** Porous Aluminum fabricated by the selective dissolution of the In phase from the Al-In alloy solidified under a magnetic field.

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