

## Formation of fine skeletal Co–Ag by chemical leaching of Al–Co–Ag ternary alloys

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### Abstract

The formation of skeletal silver–cobalt structures was attempted by means of chemical leaching of Al atoms from various ternary Al–Ag–Co alloys which were prepared by rapid solidification or mechanical alloying. Almost all Al atoms were removed for rapidly solidified alloys and their crystallographic structure was changed from fcc of Al into fcc of Ag during chemical leaching. The microstructures of the leached specimens were observed by transmission electron microscopy. TEM observation showed that the leached specimens had a skeletal structure composed of finely distributed particles and fine pores. The particles were classified into two groups by size. The first one was rather large and about 30 nm in size. The other was fine and a few nanometers. No detectable diffraction peaks corresponding to the Co phase were observed but they were detected after annealing at 1073 K for 3.6 ks. The saturation magnetization of the leached specimens gradually increased with increasing temperature and approached to the maximum. A part of this variation was due to the precipitation of Co from the supersaturated Ag(fcc) phase. The chemical leaching of Al–Co–Ag alloys might open the possibility to form a skeletal material with a metastable state in which a few cobalt atoms dissolved into the silver phase.

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### 1. Introduction

The chemical leaching process, which has been widely used for obtaining Raney catalysts [1–3] may be an attractive process to produce a skeletal metastable material with ultra fine pores. The present authors have reported some examples of how metastable phases can be formed by this process [4–8]. Also, there are some papers on amorphous or metastable phase formation by applying this process to Al–Ni and Al–Co alloys [9–12].

In this paper, we will examine the possibility to obtain skeletal Co–Ag materials by chemical leaching from various

Al–Co–Ag ternary alloys. It turned out that the Co–Ag binary system has no miscibility in both the liquid and solid states [13]. Since it has a tendency to separate into two phases above the liquidus temperature or at room temperature, it is difficult to obtain a solid solution of Co–Ag or a finely distributed structure by conventional solidification processes. However, we have proposed chemical leaching as a new process to form a metastable solid solution or a finely distributed structure [8].

The leaching process can be considered as a sort of the phase transformation induced by changing the alloy composition instead of changing the temperature as in ordinary phase transformations. If it were applied to a single phase precursor of an Al–Co–Ag ternary alloy, cobalt and silver atoms will rearrange and form a new crystal lattice by the removal

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of aluminum atoms from the original lattice sites. The newly formed structure will depend on the rearrangement process. However, there are no papers on this subject. In a previous paper [14], we attempted to prepare a single phase ternary alloy as a precursor by using various processes.

In this paper, we examined the phase transition by chemical leaching of various Al–Co–Ag ternary alloys and measured the variation of the saturated magnetization of the leached specimens.

## 2. Experimental procedure

### 2.1. Chemical leaching

The ternary Al–Co–Ag alloys listed in Table 1 were prepared by various methods as described in our previous paper [14] and used as precursor powders. The leaching was carried out in a basic solution of 20 mass% NaOH, at 333 K for 1.8 ks to remove Al atoms from the lattice sites. This is a conventional method for obtaining Raney copper catalysts [4]. The chemical reaction of Al in the precursor powders with the basic solution is the following.



The leaching was done under N<sub>2</sub> atmosphere to avoid oxidation. The concentration of the NaOH solution in this experiment was 10 times higher than the stoichiometric quantity required in the above reaction. After leaching, the specimen was washed by degassed and distilled water several times. Some of the samples were annealed at given temperatures in vacuum for various periods.

### 2.2. Characterization of structure and magnetic measurement

The structures of the leached specimens were examined by using X-ray diffraction (XRD), SEM and TEM. For TEM observation, leached powders were molded in a plastic resin. After polymerization, it was cut into a very thin slice by using an ultra microtome with a diamond knife. The chemical composition of the specimens was evaluated by ICP analysis. The magnetic properties were measured in a vibrating sample magnetometer (VSM).

Table 1  
Nominal composition of Al–Co–Ag ternary alloys (at.%)

No.	Al	Co	Ag
1	75	5	20
2	75	7	18
3	75	10	15
4	75	12.5	12.5

## 3. Results and discussion

### 3.1. Crystal structure transition obtained by chemical leaching

Fig. 1 shows the X-ray diffraction patterns of Al<sub>75</sub>Co<sub>5</sub>Ag<sub>20</sub> precursors produced by the various processes as described in a previous paper [14]. It shows that several phases existed in the precursors except in a rapidly solidified specimen (RWA). A single fcc(Al) phase was only detected in the rapidly solidified specimen. It means that both cobalt and silver atoms were dissolved into the fcc(Al) phase without the formation of any other phase. Hence, these atoms could randomly occupy the lattice sites of the supersaturated fcc(Al) phase according to the atomic composition ratio of the alloy. On the other hand, some of the silver and cobalt atoms may occupy the lattice sites of either the Ag<sub>2</sub>Al phase or the Al<sub>9</sub>Co<sub>2</sub> phase for the slowly solidified precursor (CAST) or the mechanically alloyed precursor (MA). In this case, the rearrangement of these atoms during leaching will independently be occur in each phases and it will be quite different with that from the supersaturated fcc(Al) phase. It is difficult for the slowly solidified or mechanically alloyed material to expect the formation of a metastable solid solution or finely distributed structure. A supersaturated single phase is desirable for obtaining a metastable solid solution or finely distributed structure. Therefore, in this experiment, the rapidly solidified precursors were mostly used.

Fig. 2 shows the XRD patterns obtained by leaching the precursors shown in Fig. 1. Broad peaks of a single fcc phase were observed in a specimen leached from the rapidly solidified precursor. However, the AlAg<sub>2</sub> phase was dominant in a specimen leached from the slowly solidified precursor. Both the Ag<sub>2</sub>Al and fcc phases were observed in a specimen leached from the mechanically alloyed precursor.

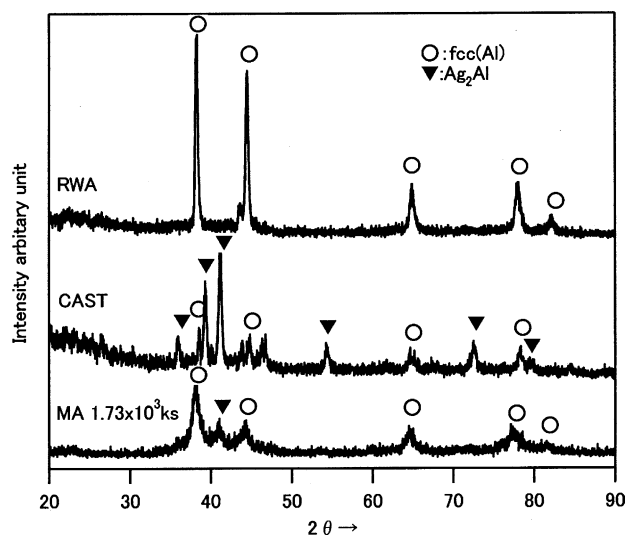


Fig. 1. X-ray diffraction patterns of variously processed precursors of Al<sub>75</sub>Co<sub>5</sub>Ag<sub>20</sub> alloy.

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