DETERMINATION OF INTERMETALLIC COMPOUNDS AND SOLID SOLUTION IN ALUMINUM ALLOYS USING ICP-AES AFTER CHEMICAL ISOLATION WITH PHENOL

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ABSTRACT After dissolving aluminum alloys in phenol, we separated insoluble intermetallic compounds from the matrix by filtration. The contents of the intermetallic compounds and the solid solutions were determined by inductively coupled plasma atomic emission spectrometry (ICP-AES). The total values of the intermetallic compounds and the solid solutions were almost equal to those of the bulk in case of Al-Mn, Al-Mg and Al-Mg-Si alloys. The amounts of solid solutions of silicon increased linearly with the tensile strength in the case of Al-Mn alloys. We found a correlation between amounts of iron of intermetallic compound and mechanical properties in the case of Al-Mg alloys. On the whole, a decrease in amounts of iron in intermetallic compounds tends to decrease both tensile and yield strengths. In the case of Al-Mg-Si samples, the results of analysis showed the form of additives varied with the heat treatment.

Keyword: *aluminum alloy, intermetallic compounds, solid solution, ICP-AES, phenol.*

1.INTRODUCTION

Form of added elements in the matrixes of aluminum alloys vary with their composition and manufacturing process, such as heat treatment, hot-rolling, cold-rolling, etc. The intermetallic compounds may decrease their sizes or dissolve in the matrix, while the dissolved components in the solid solutions may deposit by those treatments. The amount of solid solutions and intermetallic compounds in the matrixes closely related to the mechanical properties of aluminum alloys.

A few studies reported [1,2] , they referred to solid solution or intermetallic compounds separately in the above mentioned alloys. For the quantitative evaluation of them, they had to develop a method with which the matrix was selectively dissolved to get intermetallic compounds as a residue.

We tried to determine both solid solution and intermetallic compounds in aluminum alloys.

2.EXPERIMENTAL PROCEDURE

Specimens are prepared from our commercial wrought alloy strip and experimental alloys.

Intermetallic compounds were isolated from the matrix using difference of their reactivity with phenol. After dissolving matrix in phenol, intermetallic compounds were separated by filtration. Composition of the solid solutions in the filtrate and the intermetallic compounds in the residue were determined by ICP-AES. The standard for ICP-AES to determine solid solution were used that dissolved organonmetallic compounds in benzyl alcohol. The intermetallic compounds were decomposed with hydrogen fluoride and nitric acid. About metallic silicon in the residue, which dose not easily decompose, we confirmed the sufficient recovery by a test using silicon wafer. For evaluating the precision and accuracy of this method, the total values of solid solution and intermetallic compounds were compared with composition of that bulk. From the assay of Al-Mn, Al-Mg, Al-Mg-Si alloys, the relationship of form of components with the mechanical properties and heat treatment was discussed.

The solid solution and the intermetallic compounds in aluminum alloys were determined by ICP-AES(Seiko Instruments Inc., Type SPS4000). We used organic solvent system for determination of solid solution and hydrogen fluoride system for determination of intermetallic compounds. The dissolution of samples were accomplished in a separable flask with a reflux condenser. The filtration of undissolved residues with hot phenol were carried out using a polyfluoric vinylidene with pore size 0.1 μ m.

The phenol was used as a solvent, and the benzyl alcohol was used to prevent solidification of phenol. Compounds used for the standard solutions for ICP-AES to determine the solid solutions, were magnesium cyclohexanebutyrate, copper cyclohexanebutyrate, manganous cyclohexanebutyrate, ferric bezoylacetonate and octaphenylcyclotetrasiloxane. The solution for calibration had matched up to sample solution with high purity aluminum [99.999%(m/m)] .

After the phenol(ca.50ml) in the separable flask was freed from dissolved water by heating to boiling point(ca.180℃), aluminum sample was added to the flask and keep heating until it dissolved completely. The under face of cover of separable flask and inner side of separable flask were washed with benzyl alcohol. The solution was filtered though the filter with pore size 0.1 μ m and the filtrate was filtered with the same filter. The residue was washed with benzyl alcohol at several times. The filtrate was diluted to the mark of measuring flask(250ml) with benzyl alcohol. Magnesium, copper, manganese, iron and silicon in that solution were determined with ICP-AES.

The filter contained residue which obtained through 2.2.1 procedure was decomposed with 10ml of water, 2ml of sodium chloride solution(5g/100ml), 5ml of hydrogen nitrate(7mol m^{-3}) and 2ml of hydrogen fluoride(14mol m^{-3}) by heating in the Teflon beaker. After decomposing the residue on the filter, the filter was washed with water and removed from that beaker. The solution was transferred to the measuring flask(100ml) that made from polyethylene and diluted to the mark with water. Magnesium, copper, manganese, iron and silicon in the solution were determined using ICP-AES.

3RESULTS AND DISCUSSION

The silicon in aluminum alloys is known to have several forms such as Si, α-AlFeSi, Mg_2Si, etc. Among them , Si is most difficult to decompose by mineral acid. We tried to decompose the residue that obtained through the procedure of 2.2.1 with hydrogen nitrate and hydrogen fluoride. However, it seemed that silicon evaporated and lost as form of SiF_4. Sodium chloride was added into the solution for decomposition to inhibit producing of SiF_4. To evaluate, the accuracy and precision of this method, we tested using silicon wafer. The recovery of silicon were 98～103%(n=7, σ=2%) by the procedure described above.

Table 1 shows the results of analysis Al-Mn alloys with the procedure described above. The total values of the solid solutions and the intermetallic compounds were almost equal to that of the bulk. When the weight of sample was 0.10g in the case of Al-Mn alloys, the solid solution of silicon and that of iron were very small. Therefore, for Al-Mn alloys, we took 2.0g of sample.

Table1 Determination of solid solution and intermetallic compounds in Al-Mn alloys

Sample	Phase	Content of elements %(m/m)				
		Mg	Cu	Mn	Fe	Si
A	Solid solutions	1.14	0.016	0.171	0.0057	0.0043
	Intermetallic compounds	0.085	0.187	0.877	0.377	0.160
	Total	1.22	0.203	1.05	0.383	0.164
	Bulk contents	1.18	0.202	1.05	0.399	0.162
B	Solid solutions	1.23	0.021	0.185	0.018	0.037
	Intermetallic compounds	0.054	0.229	0.824	0.393	0.252
	Total	1.28	0.250	1.01	0.411	0.289
	Bulk contents	1.28	0.244	1.02	0.427	0.283

The samples of several compositions of Al-Mn alloys were manufactured at the various heating. We compared the results of these sample with mechanical properties. As Fig.1 shows, it is found that the amounts of solid solution of silicon increased linearly with the tensile strength.

We tried to make Al-Mg alloys that contents iron from 0.031%(m/m) to 1.3%(m/m). The results of these samples are in Table 2. Almost all of added iron were determined as the precipitates. The manganese and copper in intermetallic compounds increased with increasing iron. This tendency means that the manganese and copper were included into the intermetallic compounds of iron. This results were compared with tensile strength of test prepared manufactured materials. As shown in Fig.2, a decrease in amounts of iron in the intermetallic compounds tends to decrease the tensile strength and yield stress. However if increasing of iron, it did not to relate to the elongation of that alloy.

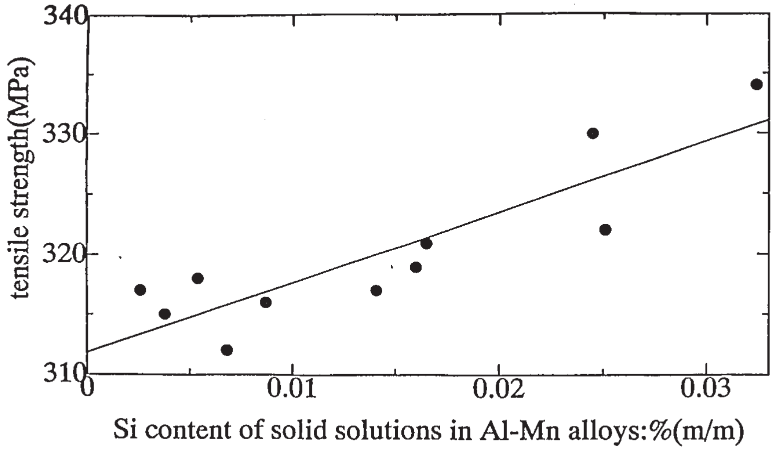

Fig.1 The relation between tensile strength and silicon content of solid solution in Al-Mn alloys

Table2 Determination of intermetallic compounds in Al-Mg alloys added iron

Sample No.	Bulk content Fe %(m/m)	Content of intermetallic compounds %(m/m)				
		Mg	Cu	Mn	Fe	Si
1	0.031	0.0815	<0.005	0.0382	0.0323	0.0523
2	0.058	0.0788	<0.005	0.0492	0.0532	0.0444
3	0.11	0.0843	0.0058	0.0683	0.112	0.0822
4	0.22	0.0818	0.0087	0.0873	0.209	0.0549
5	0.40	0.0894	0.0122	0.113	0.391	0.0396
6	0.58	0.0856	0.0177	0.123	0.561	0.0546
7	0.94	0.0814	0.0158	0.136	0.923	0.0415
8	1.3	0.0795	0.0202	0.148	1.27	0.0503
Bulk content %(m/m)		4.5	0.035	0.22	——	0.060

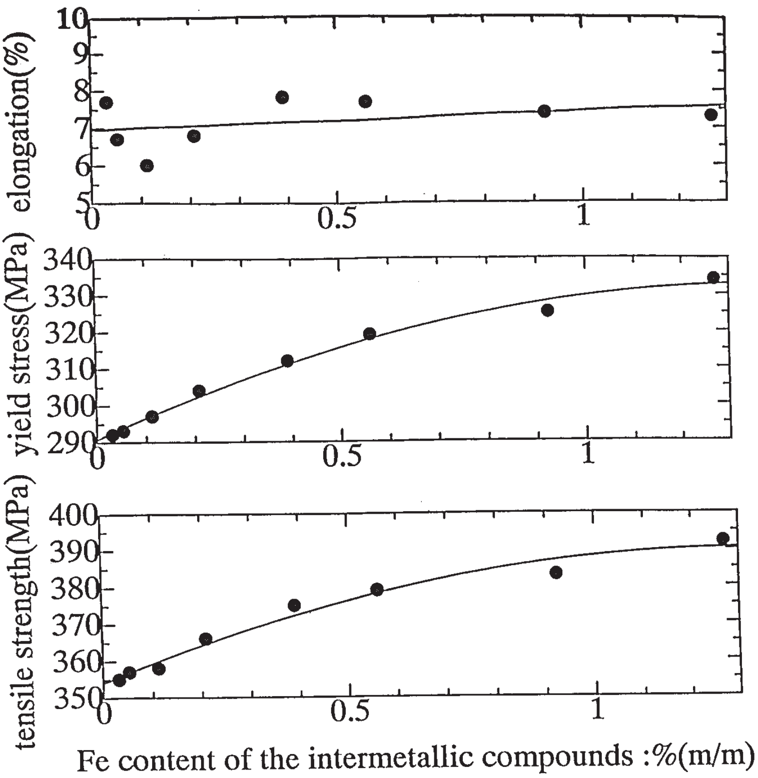

Fig.2 The relation between mechanical properties and iron content of intermetallic compounds in Al-Mg alloys

Next, we made Al-Mn alloys whose bulk content of Si we have selected 3 steps silicon. The results of these samples were shown in Table 3. The almost all of silicon was determined as the intermetallic compounds. The intermetallic compounds including magnesium were increased by means of adding silicon. This results suggested that the magnesium and silicon were formed as Mg_2Si.

Table 3 Determination of intermetallic compounds in Al-Mg alloys added silicon

Sample	Bulk content of silicon %(m/m)	Content of intermetallic compounds %(m/m)				
		Mg	Cu	Mn	Fe	Si
A	0.061	0.146	<0.005	0.0556	0.191	0.0592
B	0.13	0.208	<0.005	0.0598	0.196	0.0964
C	0.21	0.360	<0.005	0.0536	0.184	0.192
Bulk content %(m/m)		4.5	0.010	0.22	0.23	—

The Al-Mg-Si alloys including magnesium [0.59%(m/m)], silicon [0.57%(m/m)], copper [0.13%(m/m)], manganese [0.075%(m/m)] and iron [0.18%(m/m)] were made. Sample A was given the treatment of solution and the quenching. After treating the same way of sample A, sample B was given the heating treatment at 180℃ for 8hr. After treating the same way of sample A, sample C was given heating treatment at 360℃ for 2hr. The results of these samples were shown in Table3. Generally, magnesium, copper and silicon were varied on the phase. This results suggested that the amounts of intermetallic compounds such as Mg_2Si, $Cu_2Mg_8Si_6Al_5$, $CuMgAl_2$, Si, etc. were varied by means of these treatments. We are now trying to confirm this results by X-ray analysis investigation.

Table 4 Determination of solid solution and intermetallic compounds in Al-Mg-Si alloys

Sample	Phase	content of element %(m/m)				
		Mg	Cu	Mn	Fe	Si
A	Solid solutions	0.575	0.108	0.0325	0.0184	0.468
	Intermetallic compounds	<0.001	0.0223	0.0432	0.163	0.0856
	Total	0.575	0.130	0.0757	0.181	0.554
B	Solid solutions	0.557	0.0477	0.0306	0.0086	0.0264
	Intermetallic compounds	0.0144	0.0860	0.0470	0.175	0.313
	Total	0.571	0.134	0.0776	0.184	0.577
C	Solid solutions	0.207	<0.002	0.0305	0.0135	0.0724
	Intermetallic compounds	0.386	0.129	0.0433	0.164	0.492
	Total	0.593	0.129	0.0748	0.177	0.564
Bulk content %(m/m)		0.584	0.126	0.0750	0.173	0.564

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