Aging Characteristics and Weld-solidified Structure in Al-Zn-Mg Alloy Welds

By

Masao KIKUCHI* and Masao MIZUNO**

(Received September 29, 1981)

Abstract

The aging characteristics and the weld-solidified structure of weld Al-Zn-Mg alloys, and the effects of grain and dendrite cell sizes on the aging characteristics of rapidly solidified Al-Zn-Mg alloys were examined by hardness measurements, the X-ray small-angle scattering and by optical- and transmission electron microscopies.

Differences in macrostructures in weld Al-Zn-Mg alloys have no effect on their aging characteristics. Differences between microstructures of weld alloys and those of heat-affected zones in the alloy welds affect their aging characteristics. Sub-grains in the weld metals of Al-Zn-Mg alloy welds decrease their age-hardening capacities at 80°C, but increase them at 150°C. These results are discussed in terms of solute segregations and preferred precipitation η' phases at sub-grain boundaries or on dislocations in the weld metals of Al-Zn-Mg alloy welds.

1. Introduction

Weld metals are different from base metals in many respects. They have structures characterized by rapid solidification, such as their crystal form, a dendrite structure, a segregation of solute atoms, and lattice defects. In dealing with the aging phenomenon of weld metals, we have to consider these structural characteristics. So far, there have been several reports regarding the relationship between weld-solidified structures and the mechanical properties of aluminium alloys[1),2)]. There has been, however, no detailed investigation on the relationship between weld-solidified structures and their aging phenomena.

In this present research, the relationship between aging characteristics and weld-solidified structures of Al-Zn-Mg weld alloys, has been studied by hardness measurements, the X-ray small-angle scattering, and by optical- and transmission electron microscopies. The effects of both grain- and dendrite cell sizes on the aging characteristics of rapidly solidified Al-Zn-Mg alloys were also investigated.

---

* Research Student.

** Department of Metal Science and Technology.

2. Experimental Method

An Al-Zn-Mg alloy was prepared by melting highly pure Al, Zn and Mg. Four other Al-Zn-Mg alloys containing small amounts of Ti and B were also prepared. The chemical compositions of these alloys are shown in Table 1.

Table 1. Chemical compositions of materials (wt.%).

No.	Zn	Mg	Fe	Ti	B	Al
1	4.48	1.16	<0.01	<0.01	<0.001	Re.
2	4.22	1.17	0.02	<0.01	<0.001	Re.
3	4.24	1.20	0.02	0.01	0.001	Re.
4	4.19	1.16	0.02	0.01	0.002	Re.
5	4.29	1.17	0.02	0.02	0.002	Re.

The melting was carried out in the longitudinal direction of the middle part of the plate materials ($6^t \times 150 \times 300$ mm), without using a filler metal, by means of the semi-automatic TIG welding apparatus, with a direct current straight polarity. After melting, the test materials were cooled to room temperature. The welding conditions are shown in Table 2.

Table 2. Welding conditions for TIG arc melting.

Arc Voltage (V)	Welding Current (A)	Welding Speed (mm/min)
13.0~15.0	260~270	200

Alloy No. 1 was melted in an alumina crucible in an Ar atmosphere, and then cast into a water-cooled copper mold at 720°C. The mold used for casting consisted of two copper parts and three plaster parts, as is shown in Fig. 1. The cooling rate during solidification was changed by adjusting the gap distance between the two copper parts. The cast materials were cooled to 500°C, and then placed in ice water.

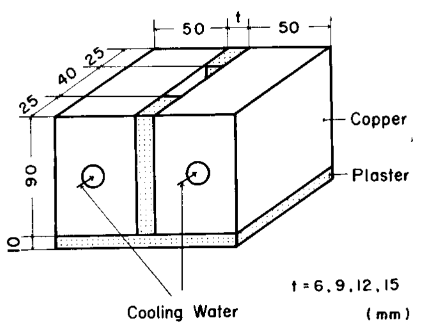

Fig. 1. Schematic illustration of casting mold.

Before the measurements, the aging treatments were carried out in an oil bath at 80°C and 150°C.

For hardness measurements, a micro Vickers hardness tester was used, at loads of 300 g and 500 g. The measurements of the X-ray small-angle scattering were carried out using Ni-filtered CuK_α radiations. The scattering intensity was counted at room temperature at (1/8)° intervals in the scattering angle (2θ) up to $2\theta_0=4°$ with line collimation. The observed spectra were corrected for their back-grounds, using pure aluminium foil with nearly the same thickness as the specimen. The calculation of the integrated intensity (Q_0) was carried out by a method developed by Gerold[3]. The average precipitate radius (R_G) was estimated by using Guinier's approximation[4]. Thin foils prepared by electrolytic polishing in a 9 % solution of perchloric acid in ethyl alcohol were examined with a transmission electron microscope.

3. Experimental Results

3.1 Aging Characteristics and Macro- and Microstructures of Weld Al-Zn-Mg Alloys

The macrostructure of a melted bead (hereafter referred to as a weld metal), obtained by melting the Al-Zn-Mg alloy plate (No. 1) with a TIG arc is shown in Photo. 1. Macroscopically, the weld metal consists of columnar crystals facing

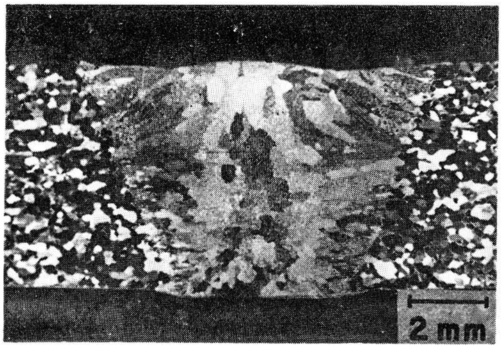

Photo. 1. Macrostructure of the weld metal in the Al-4.5%Zn-1.2%Mg alloy weld.

the fusion line towards the central part, and equiaxed crystals in the central part. In order to elucidate the relationship between this macrostructure of the weld metal and the aging characteristics, the hardness distribution of the weld metal was measured. Fig. 2 (a), (b) and (c) shows the results obtained immediately a) after-welding, b) after-aging at 80°C for 300 days and c) after-aging at 150°C for 7 days respectively. These illustrations clearly indicate that in the weld metal,

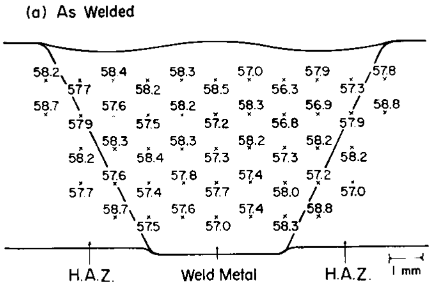

Fig. 2(a).

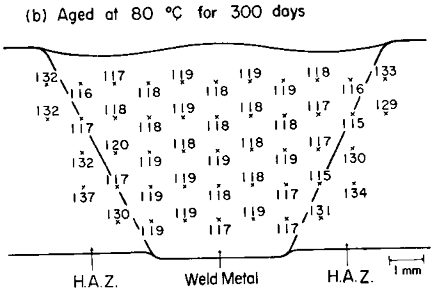

Fig. 2(b).

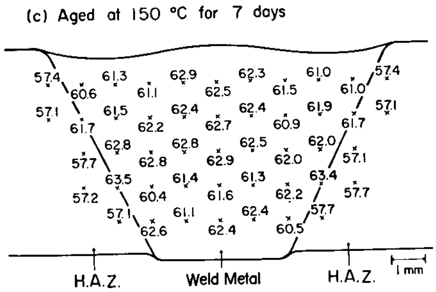

Fig. 2(c).

Fig. 2. Hardness distributions of weld metal in the Al-4.5%Zn-1.2%Mg alloy weld: (a) as welded, (b) aged at 80°C for 300 days and (c) aged at 150°C for 7 days.

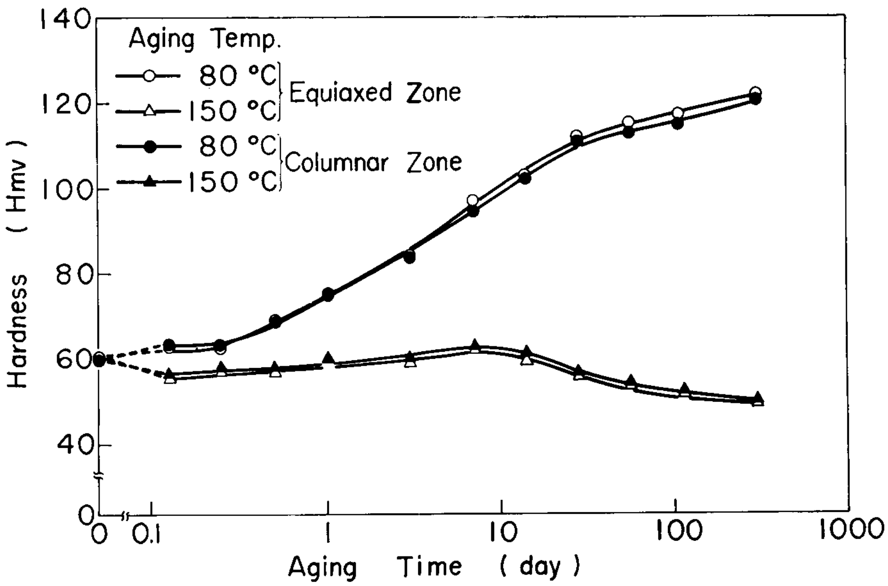

Fig. 3. Hardness vs. aging time curves for the equiaxed zone and those for the columnar zone in the weld metal of the Al-4.5%Zn-1.2%Mg alloy weld, aged at 80°C and 150°C after welding.

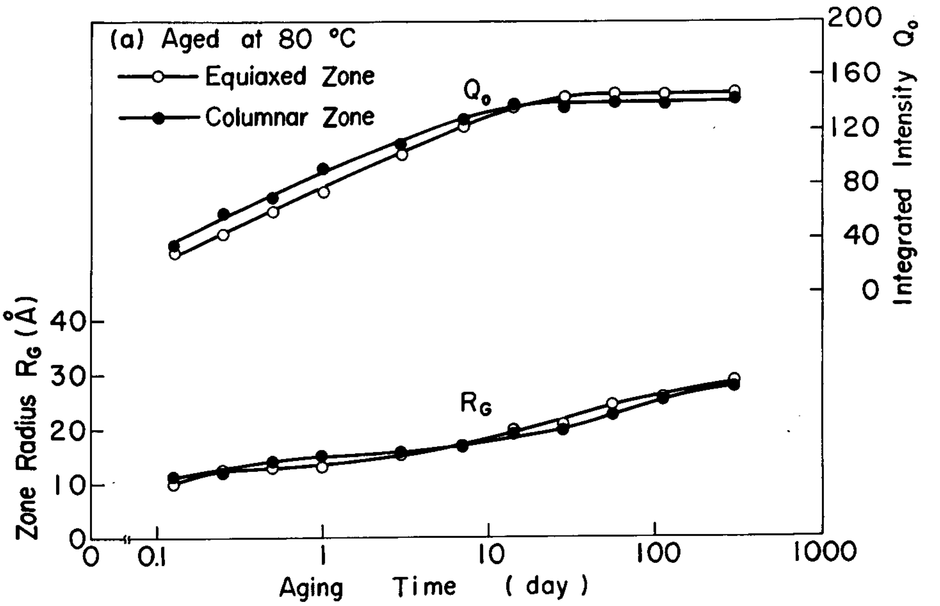

Fig. 4(a).

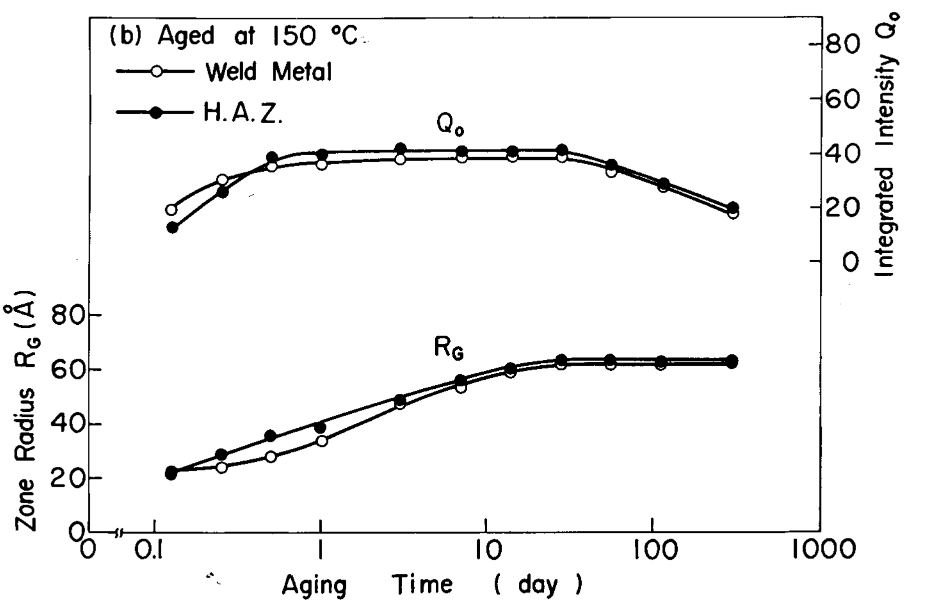

Fig. 4(b).

Fig. 4. Changes in the average zone radius and the X-ray integrated intensity of the equiaxed zone and those of the columnar zone in the weld metal of the Al-4.5%Zn-1.2%Mg alloy weld, during aging at (a) 80°C and (b) 150°C after welding.

the hardness value is almost constant in all three cases, and that differences in the macrostructure do not have any effect on the hardness distribution.

Next, in order to make a detailed investigation of the relationship between the aging process of the weld metal and the macrostructure, the weld metal was divided into two regions, namely the equiaxed crystal part and the columnar crystal part. The hardness (Hmv), the precipitate radius (R_G) and the integrated intensity (Q_0) in these two regions were measured as a function of aging time. The hardness-time curves of these two regions for 80°C aging as well as 150°C aging are compared in Fig. 3. No difference is found between the changes in hardness in the equiaxed crystal part and those in the columnar crystal part. Fig. 4 (a) and (b) shows the changes in R_G and Q_0 in these two regions for 80°C and 150°C agings. As in the case of hardness, almost no difference in R_G and Q_0 is found during aging between the equiaxed crystal part and the columnar crystal part. Therefore, it becomes clear that differences in macrostructure have practically no effect on the aging characteristics of the weld metal.

The microstructure of the weld metal is shown in Photo. 2. As indicated in the top left-hand picture, which is a macrophotograph, A, B and C show the structures in the equiaxed crystal part, in the columnar crystal part and in the fusion boun-

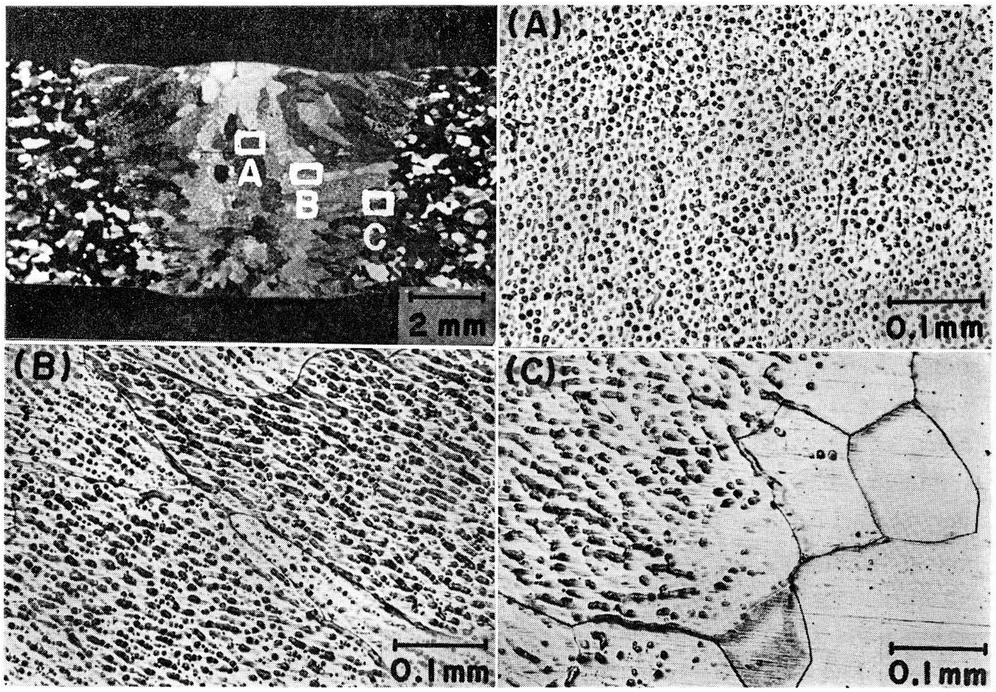

Photo. 2. Microstructure of the weld metal in the Al-4.5%Zn-1.2%Mg alloy weld.

dary part, respectively. Microscopically, within the various structures of the weld metal, sub-grains (cells, cellular dendrites or equiaxed dendrites) are formed. In the fusion boundary part, crystal grains are seen of which a part has been melted and solidified. In these crystal grains, there is a region in which sub-grains have been formed, together with a region in which sub-grains have not been formed.

The characteristic feature of the microstructure of the weld metal will be represented by these sub-grains. Therefore, in order to investigate the relationship between the aging characteristics and the microstructure of the weld metal, attention was paid to the partly melted and solidified crystal grains in the fusion boundary part. In these crystal grains, we have two regions: the melted and solidified regions in which sub-grains have been formed (weld metal), and the non-melted regions with no sub-grains (heat-affected zone). Therefore, by comparing the aging processes in these two regions, we will obtain the relationship between the sub-grains and the aging characteristics in a weld metal.

The hardness values on the weld metal and heat-affected zone sides in these partly melted and solidified crystal grains were compared immediately after welding, after aging at 80°C for 300 days and after aging at 150°C for 7 days. The results are shown in Photo 3: (a), (b) and (c), where the dark rectangles are indentations for the hardness measurements, while the numerical values show the respective hardness values. The hardness values on the weld metal side and on the heat-

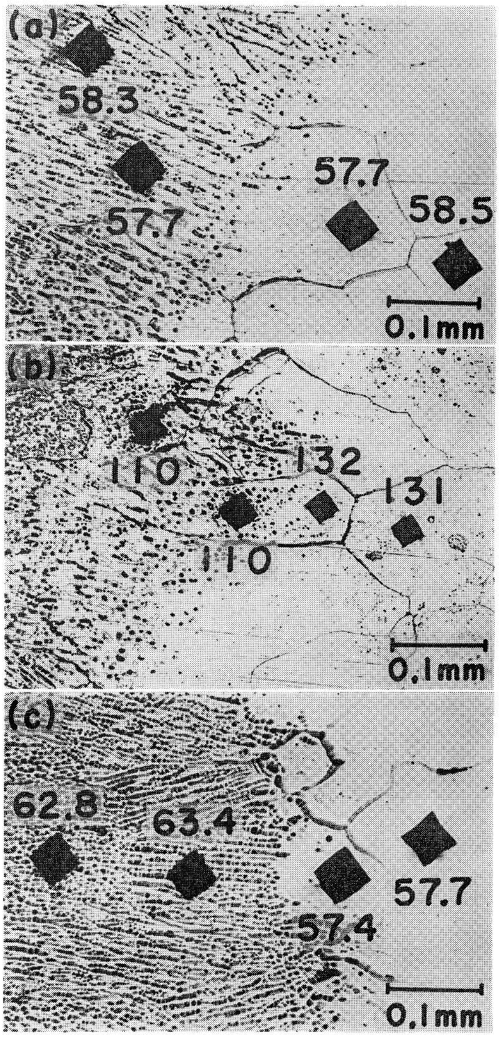

Photo. 3. Hardnesses of the weld metal and those of the heat-affected zone in the Al-4.5% Zn-1.2%Mg alloy weld: (a) as welded, (b) aged at 80°C for 300 days and (c) aged at 150°C for 7 days.

affected zone side in the partly melted and solidified crystal grains are nearly the same as the hardness values in the adjacent crystal grains.

Therefore, the values in the two regions in these partly melted and solidified crystal grains can be used as representative values of the hardness in the weld metal and in the heat-affected zone. Immediately after welding, the hardness values on the weld metal side are approximately the same as those on the heat-affected zone side. However, after aging at 80°C for 300 days, the hardness values on the heat-affected zone side become considerably higher than those on the weld metal side. On the other hand, after aging at 150°C for 7 days, the hardness values on the weld metal side are higher than those on the heat-affected zone side.

Since aging treatments after welding give rise to differences in hardness between the weld metal region and the heat-affected zone, the aging processes of these two regions were compared. Fig. 5 shows the hardness-time curves of these

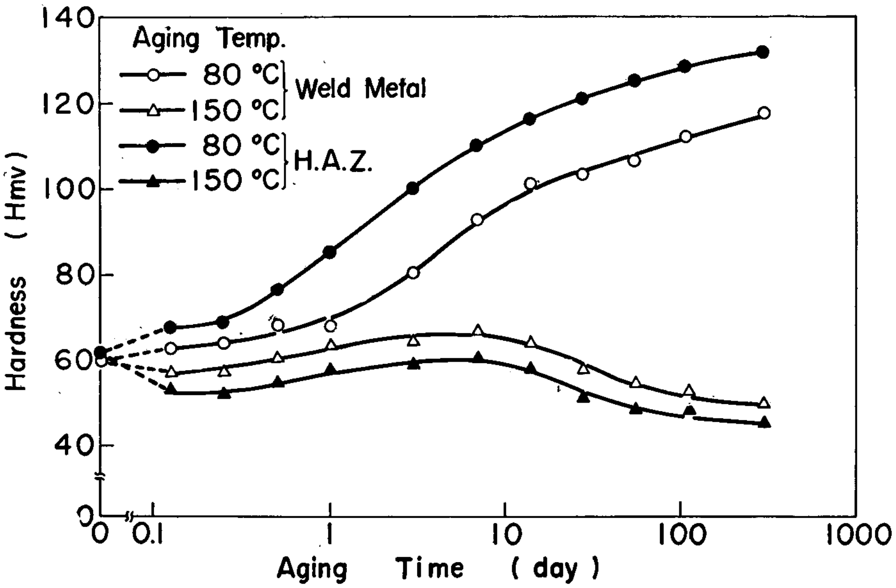

Fig. 5. Hardness vs. aging time curves for the weld metal and those for the heat-affected zone in the Al-4.5%Zn-1.2%Mg alloy weld, aged at 80°C and 150°C after welding.

two regions for 80°C aging and 150°C aging. Here, each hardness value indicates the mean value over several partly melted and solidified crystal grains in the fusion boundary part. Immediately after welding, there is almost no difference in hardness between the weld metal and the heat-affected zone. However, by aging at 80°C after welding, the hardness value of the heat-affected zone becomes higher than that of the weld metal. This difference increases with the aging time, and after 3 days it becomes approximately constant (about 20 in Vickers hardness).

The effect of 150°C aging is opposite to that of 80°C aging. The hardness value of the weld metal becomes higher than that of the heat-affected zone, although the difference is very small.

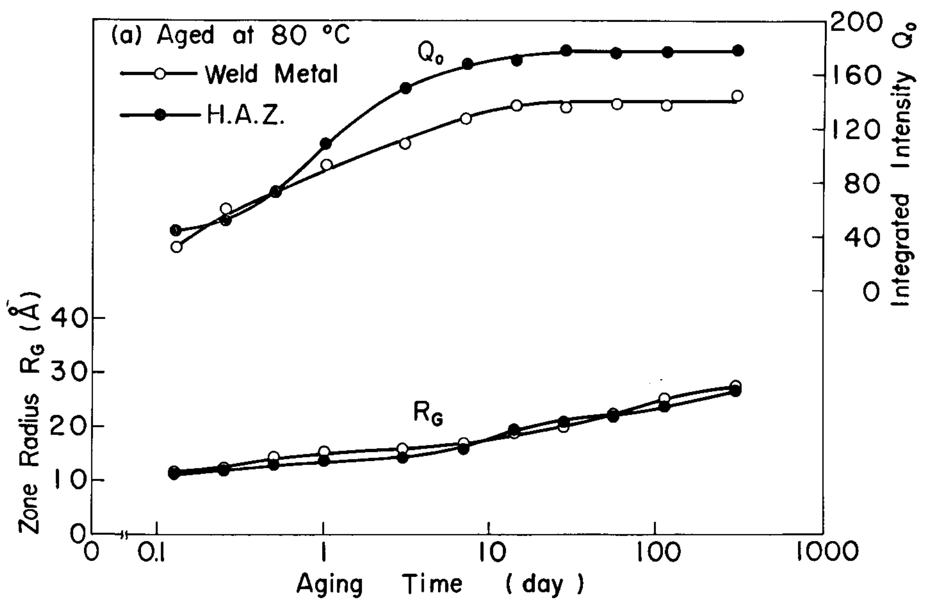

Fig. 6(a).

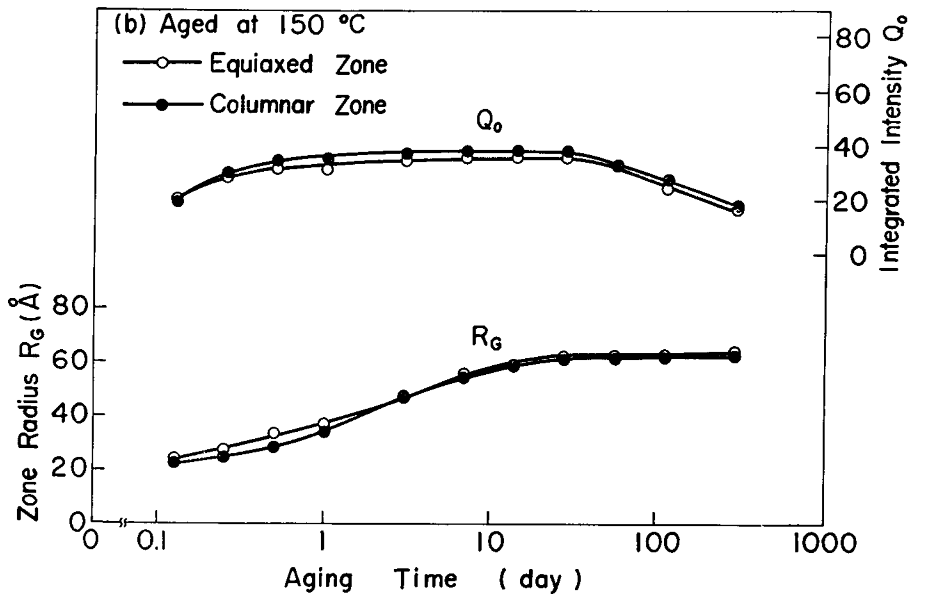

Fig. 6(b).

Fig. 6. Changes in the average zone radius and the X-ray integrated intensity in the weld metal and those in the heat-affected zone of the Al-4.5%Zn-1.2%Mg alloy weld, during aging at (a) 80°C and (b) 150°C after welding.

The values of the mean precipitate radius (R_G) and integrated intensity (Q_0) in the weld metal and the heat-affected zone in the vicinity of the fusion boundary were measured for 80°C and 150°C aging. The results are shown in Fig. 6 (a) and (b). The changes in R_G for 80°C aging reveal almost no difference between the weld metal and the heat-affected zone. On the other hand, Q_0 of the two regions have approximately the same values in the initial period of aging, whereas Q_0 of the heat-affected zone becomes larger than that of the weld metal with increasing aging time. This difference becomes largest after about 3 days aging. The values of both Q_0 increase after 3 days aging, keeping their difference constant. However, after about 30 days both values become constant. On the other hand, in the case of 150°C aging, no difference in the changes in R_G and Q_0 is found between the weld metal and the heat-affected zone. An exception is the initial period of aging, where the R_G of the heat-affected zone is slightly greater than that of the weld metal.

These results clearly show that the presence of sub-grains, which is a characteristic feature of the microstructure of the weld metal, has an effect on the aging characteristics of the weld metal.

3.2 Effects of Grain- and Dendrite Cell Sizes on Aging Characteristics of Rapidly Soldified Al-Zn-Mg Alloys

Four kinds of melted beads (weld metal) with differerent grain sizes were obtained by melting plate materials of Al-Zn-Mg alloys containing small amounts of Ti and B with a TIG arc. The microstructures of these weld metals are shown in Photo. 4 (a)~(d). It is clear that the grain size of the weld metal decreases with the increasing contents of Ti and B. The relationship between the grain size of the weld metal and the contents of Ti and B is shown in Fig. 7. No detectable difference in the dendrite cell size has been found among these weld metals.

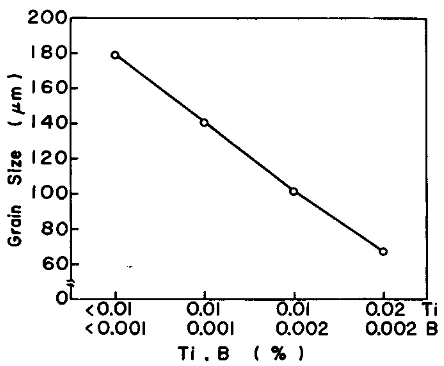

Fig. 7. Relationship between the contents of Ti and B and the grain size for the weld metal in the Al-Zn-Mg alloy weld

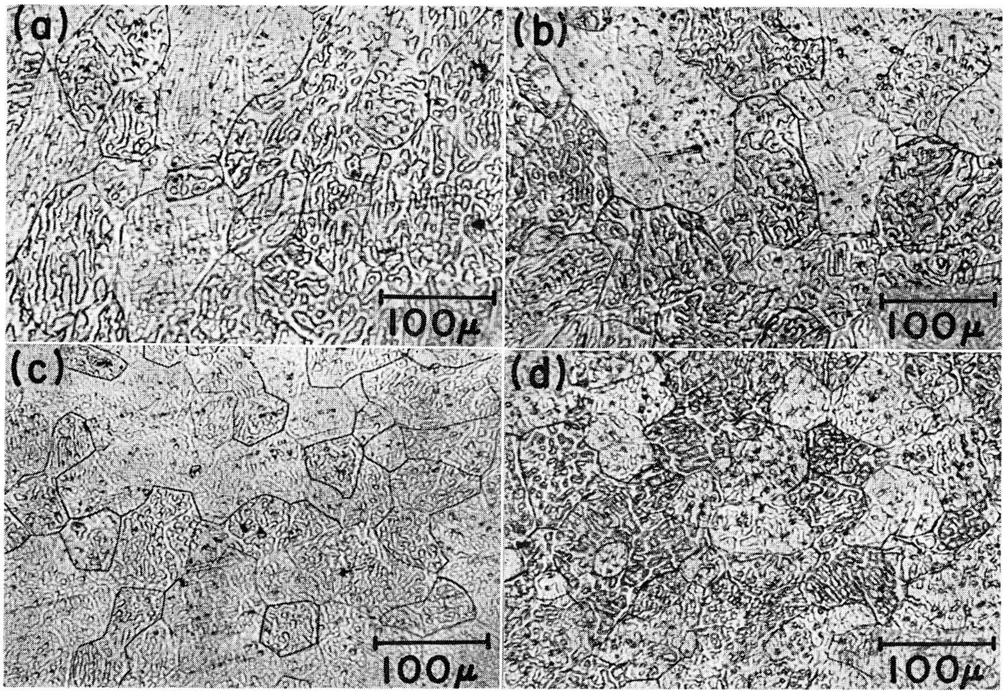

Photo. 4. Microstructures of the weld metals in the Al-Zn-Mg alloy welds: (a) No. 2, (b) No. 3, (c) No. 4 and (d) No. 5.

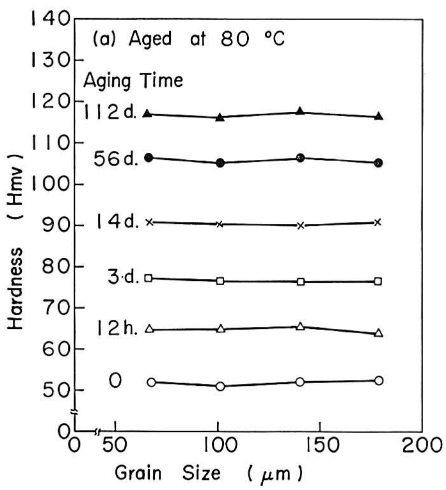

Fig. 8(a).

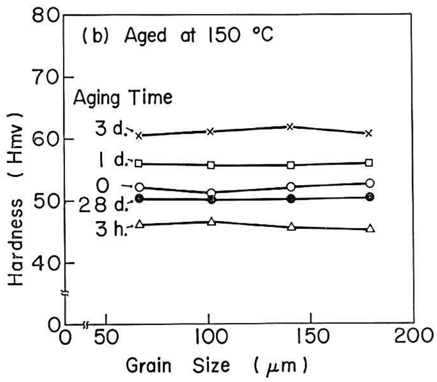

Fig. 8(b).

Fig. 8. Effects of grain size on the hardness changes during aging at (a) 80°C and (b) 150°C for the weld metal in the Al-Zn-Mg alloy weld metal.

The changes in hardness for 80°C and 150°C aging are shown as a function of the grain size of Al-Zn-Mg alloy weld metals in Fig. 8 (a) and (b), which show that the hardness values are nearly constant irrespective of the grain size at each aging time for both aging temperatures.

The changes in R_G and Q_0, corresponding to these changes in hardness, were studied by means of an X-ray small-angle scattering method. As shown in Fig. 9

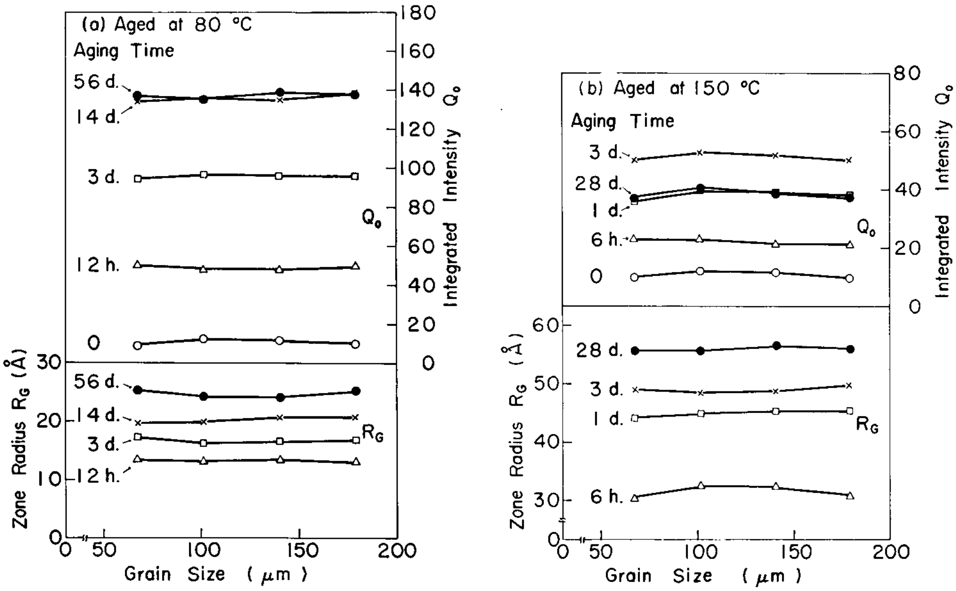

Fig. 9(a). Fig. 9(b).

Fig. 9. Effects of grain size on the change in the average zone radius and the X-ray integrated intensity, during aging at (a) 80°C and (b) 150°C for the weld metal in the Al-Zn-Mg alloy weld.

(a) and (b), R_G and Q_0 are constant against the grain size of Al-Zn-Mg alloy weld metals for both 80°C and 150°C aging. As in the case of hardness, the values of R_G and Q_0 are nearly independent of the grain size at each aging temperature and aging time.

The effects of a dendrite cell size on the aging characteristics of rapidly solidified Al-Zn-Mg alloys were studied for four kinds of cast specimens with different dendrite cell sizes. The microstructures of these cast specimens and the dependence of the dendrite cell size on the thickness of cast specimens are shown in Photo. 5 (a)~(d) and Fig. 10. It is clear that the dendrite cell size increases with the increasing thickness of the specimens.

Fig. 11 (a) and (b) show the dependence of the hardness on the dendrite cell size of rapidly solidified Al-Zn-Mg alloys for 80°C and 150°C aging. The dendrite

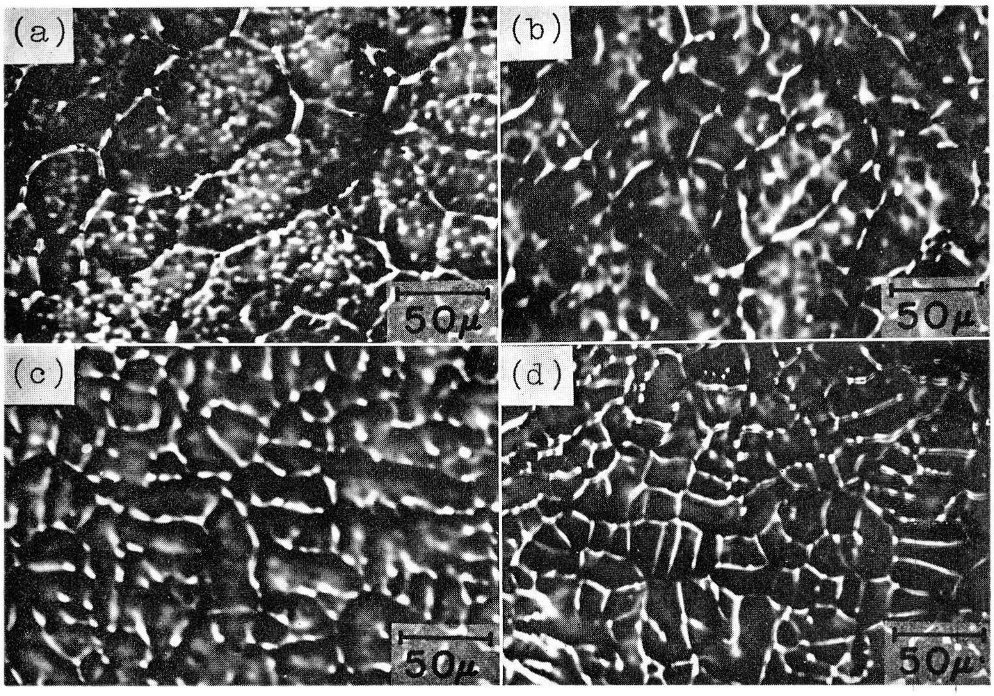

Photo. 5. Microstructures of the cast Al-Zn-Mg alloys. Specimens are (a) 15, (b) 12, (c) 9 and (d) 6 mm thick.

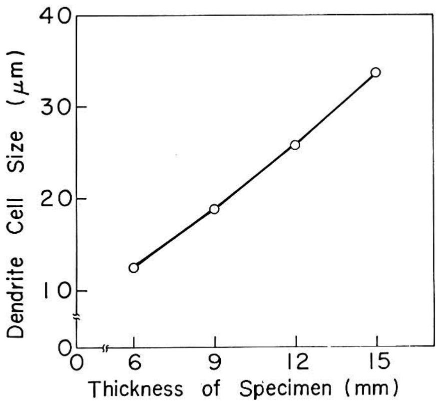

Fig. 10. Relationship between the thickness of specimen and the dendrite cell size for the cast Al-Zn-Mg alloy.

cell size has no effect on the hardness value of quenched Al-Zn-Mg cast alloys. However, the hardness value increases with a decreasing dendrite cell size of rapidly solidified Al-Zn-Mg alloys aged at 80°C after quenching. This tendency becomes more pronounced as the aging proceeds. On the other hand, in the case of aging at 150°C, such an evident tendency as in the 80°C aging has not been

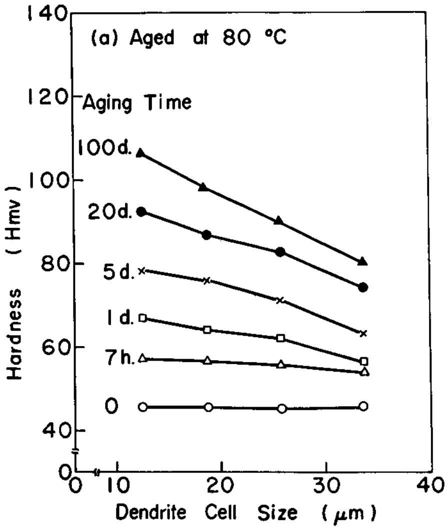

Fig. 11(a).

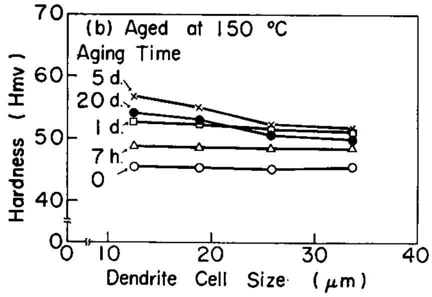

Fig. 11(b).

Fig. 11. Effects of the dendrite cell size on the hardness change during aging at (a) 80°C and (b) 150°C for the cast Al-Zn-Mg alloys.

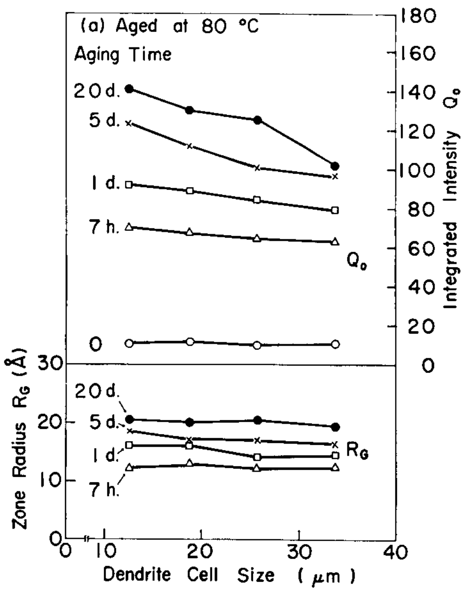

Fig. 12(a).

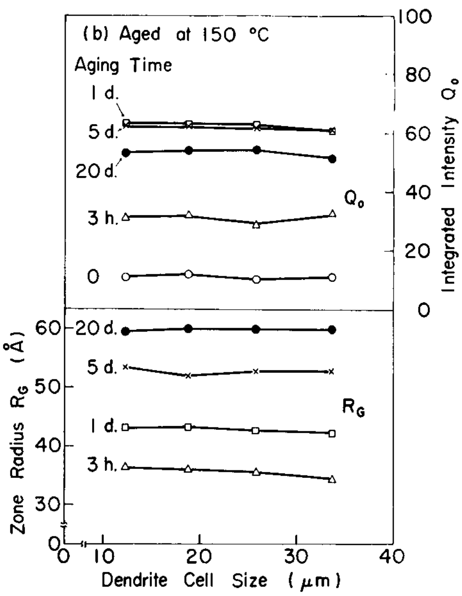

Fig. 12(b).

Fig. 12. Effects of the dendrite cell size on the change in the average zone radius and the X-ray integrated intensity, during aging at (a) 80°C and (b) 150°C for the cast Al-Zn-Mg alloy.

observed.

Fig. 12 (a) and (b) show the changes in R_G and Q_0 as a function of the dendrite cell size of rapidly solidified Al-Zn-Mg alloys when aged at 80°C and 150°C. In the case of the 80°C aging, there has been no appreciable effects of the dendrite cell size on the value of R_G. However, remarkable effects on the changes in Q_0 have been found. The value of Q_0 increases with a decreasing dendrite cell size, and this tendency becomes more pronounced as the aging proceeds. On the other hand, no effect on the changes in R_G and Q_0 is found for 150°C aging.

4. Discussion

4.1 Aging Characteristics and Macro- and Microstructures of Weld Al-Zn-Mg Alloys

In 3.1, the dependence of the aging characteristics on the weld-solidified structure of an Al-Zn-Mg alloy weld metal was discussed from the viewpoints of both the macro- and microstructures. As a result, it becomes evident that differences in the macrostructure (differences in the crystal form etc.) have no effect on the aging characteristics of the weld metal. However, the presence of sub-grains, which is a characteristic feature of the microstructure, has a great effect on these characteristics. Therefore, we will discuss the relationship between the microstructure and the aging characteristics of the weld metal.

In the latter half of 3.1, in order to know the relationship between the aging characteristics and the microstructure of the weld metal, special attention was given to the partly melted and solidified crystal grains in the fusion boundary part. By comparing the aging processes in the melted and solidified regions (weld metal) and non-melted regions (heat-affected zone) in these crystal grains, we found the relationship between the aging characteristics and the sub-grains, which is a characteristic feature of the microstructure of the weld metal. Other factors besides the microstructure, for example, the cooling rate after solidification[5] etc., can be probably ignored because these two regions exist near each other.

In a previous section we have shown that the presence of sub-grains has an effect on the aging characteristics of the weld metal. (See Photo. 3, Figs. 5 and 6.) By 80°C aging after welding, the hardness in the weld metal becomes lower than that in the heat-affected zone, and the amount of precipitates corresponding to Q_0[4] also becomes smaller in the weld metal. On the other hand, no difference has been observed between the amounts of precipitates in the weld metal and in the heat-affected zone for the 150°C aging. The hardness of the weld metal becomes higher than that of the heat-affected zone. These results are explained as follows.

Firstly, for 80°C aging, fine G.P. zones precipitate uniformly in the matrix, giving rise to a hardening in both the weld metals and the heat-affected zones. However, in a rapidly solidified structure such as weld metals, solute atoms segregate towards crystal grain boundaries as well as sub-grain boundaries during solidification[6),7)]. This leads to a lower concentration of the matrix than the average solute concentration. Therefore, the amount of precipitated G.P. zones in the weld metal becomes lower than that in the heat-affected zone (Fig. 6 (a)), and at the same

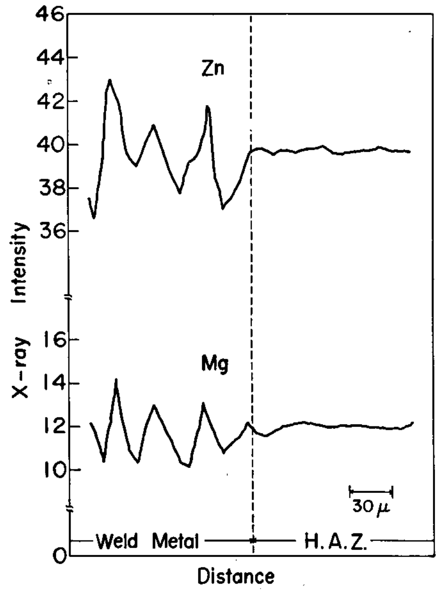

Fig. 13. Distributions of Zn and Mg atoms near the fusion line in the Al-4.5%Zn-1.2%Mg alloy weld.

time, the hardness becomes lower (Photo. 3 (b), Fig. 5). The distribution of Zn and Mg atoms in the weld metal side and the heat-affected zone side was measured by means of an X-ray probe microanalysis, as shown in Fig. 13. The Zn and Mg atoms segregate on the weld metal side, and the solute concentration of the matrix becomes lower than on the heat-affected zone side.

In the case of 150°C aging, the intermediate η' phase precipitates and contributes to the hardening. The η' phase precipitates preferentially at the grain boundaries, sub-grain boundaries and dislocations. In the weld metal, since a large number of these places, which are preferable for precipitation, are introduced during solidification, the intermediate phase is more finely dispersed and the hardness value becomes higher. The transmission electron micrographs of the weld metal and the heat-affected zone aftei aging at 150°C for 7 days (Photo. 6 (a)

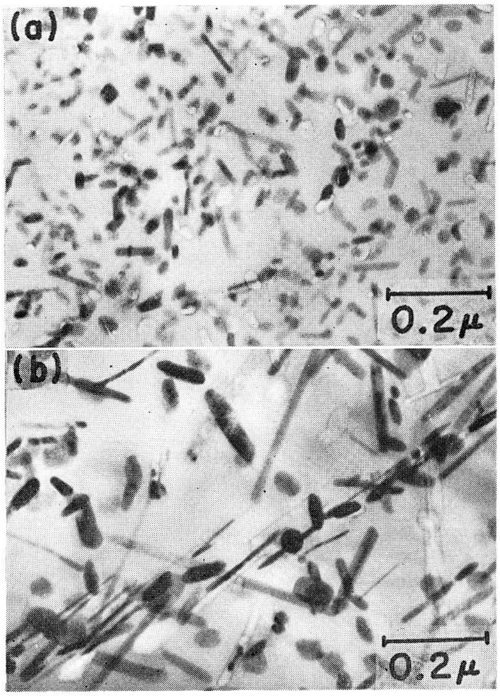

Photo. 6. Transmission electron micrographs of (a) the weld metal and (b) the heat- affected zone in the Al-4.5%Zn-1.2% Mg allloy weld, aged at 150°C for 7 days after welding.

and (b)) clearly show that the size of the precipitated phase in the heat-affected zone is considerably large, whereas that in the weld metal is fine.

4.2 Effects of Grain- and Dendrite Cell Sizes on Aging Characteristics of Rapidly Solidified Al-Zn-Mg Alloys

In 3.2, the effects of grain- and dendrite cell sizes on the aging characteristics of rapidly solidified structures have been discussed. We have shown that the dendrite cell size has remarkable effects on the aging characteristics of rapidly solidified Al-Zn-Mg alloys, while the grain size has no effect on these characteristics. Generally, in a rapidly solidified metal such as weld metals, solute atoms easily segregate towards dendrite cell boundaries[8)], leading to a decrease in the solute concentration in the matrix. The amount of segregates increases with an increasing dendrite cell size, and at the same time, the solute concentration in the matrix becomes lower[9)]. This decrease in the solute concentration of the matrix results in a decrease of the amount of fine precipitates at 80°C or 150°C aging. Hence, the hardness becomes lower as clearly shown in Figs. 11 (a) and 12 (a) for 80°C aging. Thus, the effect of the dendrite cell size on the aging characteristics of rapidly solidified Al-Zn-Mg alloys is mainly attributed to a decrease in the solute concentration in the dendrite cell matrix. In contrast to the 80°C aging, no appreciable effect of the dendrite cell size on the aging characteristics of rapidly solidified Al-Zn-Mg alloys has been observed for the 150°C aging. In this case, since the amount of precipitates and the hardness are smaller than those for 80°C aging, a variation of the dendrite cell size gives no detectable difference in Q_0 in hardness.

5. Conclusion

The dependence of the aging characteristics on the weld-solidified structure of Al-Zn-Mg alloy weld metals and the effects of grain- and dendrite cell sizes on the aging characteristics of rapidly solidified Al-Zn-Mg alloys were investigated by hardness measurements, the X-ray small-angle scattering method, and by optical- and transmission electron microscopies. The results obtained are as follows:

(1) Differences in macroscopic crystal form in the weld-solidified structure have no effect on the aging characteristics of the weld metal.

(2) The sub-grains characteristic of the microstructure of the weld-solidified structure has a remarkable effect on the aging characteristics of the weld metal.

(3) The effect of the microstructure on the aging characteristics of the weld metal depends on the aging temperature. The presence of sub-grains reduces the age-hardenability of the weld metal by the 80°C aging, whereas it increases the hardening capacity by the 150°C aging. These results are explainable by the segregations of solute atoms on sub-grain boundaries and preferential precipitations of the intermediate phase.

(4) The aging characteristics of rapidly solidified Al-Zn-Mg alloys do not depend on grain size.

(5) The dendrite cell size has an effect on the aging characteristics of rapidly solidified Al-Zn-Mg alloys. The age-hardening capacity of rapidly solidified Al-Zn-Mg alloys decrease as the dendrite cell size increases. This fact is attributed to a decrease in solute concentrations in the dendrite cell matrix, being in agreement with an increase in the dendrite cell size.

Acknowledgements

The authors wish to express their gratitude to Mrs. K. Horii and T. Morita for their kind help with the experiments in this work.

References

1) J.H. Rogerson: Aluminium, 48 (1972), 424.
2) T. Fukui: J. Japan Inst. Light Metals, 24 (1974), 180.
3) A. Guinier and G. Fournet: Small-Angle Scattering of X-rays, John-Wiley and Sons, (1955), 24.
4) V. Gerold: Phys. Stat. Sol., 1 (1961), 37.
5) M. Kikuchi, Y. Kitaguchi and M. Mizuno: J. Japan Inst. Light Metals, 26 (1976), 451.
6) F. Matsuda: Seminar Text, Japan Inst. Metals, (1969), 124.
7) A.T. D'Annessa: Weld. J., 45 (1966), 569s.
8) T.F. Bower, H.D. Brody and M.C. Flemings: Trans. Met. Soc. AIME, 236 (1966), 624.
9) T. Fukui and K. Nanba: J. Japan Inst. Light Metals, 20 (1970), 458.