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Nucleation Mechanisms and Grain Refining of Weld Metal

For the first time, heterogeneous nuclei are observed in the weld pool, and a method for identifying the nucleation mechanism is demonstrated

BY S. KOU AND Y. LE

ABSTRACT. A microstructural study was carried out to better understand one of the most important aspects of weld metal solidification, i.e., nucleation and grain refining. The microstructure in the welding zone was preserved by quenching during welding and was examined afterward. Heterogeneous nuclei in the weld pool, as well as partially melting grains along the leading portion of the weld pool boundary and growing dendrites along the trailing portion of the weld pool boundary, were clearly revealed. Based on the microstructure observed, three different mechanisms of weld metal nucleation and grain refining were discussed, i.e., dendrite fragmentation, grain detachment and heterogeneous nucleation. The interaction between weld pool convection and the microstructure around the weld pool boundary was described, and its effect on weld metal

nucleation and grain refining was discussed. A method was presented for identifying the nucleation and grain refining mechanism of the weld metal, by changing the microstructure around the weld pool boundary and dissolving heterogeneous nuclei through overlap welding, and by examining the grains of the weld metal with electron microscopy. Welds of commercial 6061 aluminum alloy were used as an example for demonstrating this mechanism identification method.

Introduction

The grain structure of the weld metal can affect significantly the soundness and properties of the resultant weld. Fine equiaxed grains tend to reduce solidification cracking (Refs. 1-3) and improve mechanical properties of the weld metal, such as toughness, ductility, strength and fatigue life (Refs. 4-6).

The grain structure near the fusion boundary of the weld metal is dominated by epitaxial growth, as first reported by Savage, et al. (Refs. 7, 8). The grain structure in the bulk weld metal, however, is dominated by competitive growth. In the absence of nucleation in the bulk weld metal, the columnar grains growing epitaxially from the fusion boundary compete with each other for space. The columnar grains which have their easy growth direction lining up favorably with the direction of maximum temperature gradient tend to grow faster and crowd out other columnar grains. When nucleation is operating, however, new grains will nucleate and grow in the bulk weld metal, blocking off the columnar grains growing epitaxially from the fusion boundary. The greater the extent of nucleation, the more numerous and finer the new grains are, i.e., the more grain refining of the weld metal.

The nucleation mechanisms and grain refining of the weld metal have been studied by a number of investigators. However, an integrated picture of the microstructure of the welding zone, which is an important key to the understanding of weld metal nucleation and grain refining, has not been provided. As

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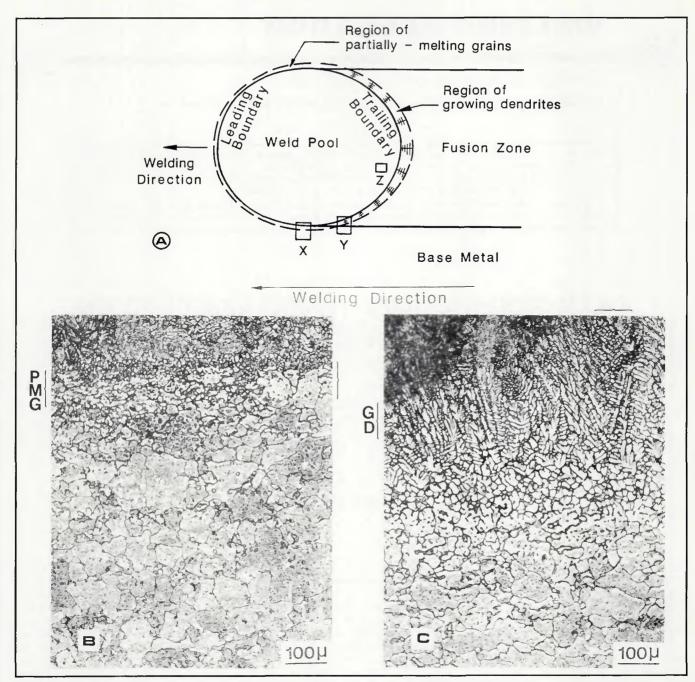


Fig. 1—Microstructure around the weld pool boundary quenched during the GTA welding of 2219 aluminum alloy. A—schematic sketch; B—micrograph taken at position X; C—micrograph taken at position Y. The brackets in B and C denote regions of partially melting grains and growing dendrites, respectively

will be described later in this paper, the mechanisms of dendrite fragmentation, grain detachment and heterogeneous nucleation can be well explained by the microstructure of the welding zone. Also, there are situations where more than one of these mechanisms are possible, and a method is needed for identifying which mechanism is responsible for the nucleation and grain refining produced in the weld metal.

This paper deals with the important subject of weld metal nucleation and grain refining, with emphasis on the microstructure of the welding zone, its connection with the mechanisms of nucleation and grain refining, and the identification of the right mechanism. The effect of welding variables (e.g., the heat input and welding speed, the oscillation and pulsation of the arc, the gas cooling of the weld pool surface, etc.) on weld metal nucleation and grain refining will be addressed in subsequent reports. The ultimate goal of this and subsequent studies is to help achieve effective control or refinement of the weld metal grain structure.

Experimental Procedure

The material used for welding was commercial 6061 aluminum sheets of 1.6-mm (1/16-in.) thickness. The nominal chemical composition of 6061 aluminum alloy is Al, 0.6Si, 1.0Mg, 0.3Cu, 0.2Cr, in wt-%. Commercial 2219 aluminum sheets of the same thickness were also used, the nominal chemical composition being Al, 6.3Cu, 0.3Mn, 0.06Ti, in wt-%.

A programmable gas tungsten arc (GTA) welding machine was used in conjunction with a 4-pole magnetic arc oscil-

lator for oscillated arc welding. Quenching of the weld zone was carried out by introducing ice water from behind the torch. This allowed the arc to be extinguished instantaneously and the microstructure of the welding zone during welding to be preserved.

The test welds, *i.e.*, the welds for which the nucleation mechanism of fine equiaxed grains was to be determined, were made with circular arc oscillation at the frequency of 35 Hz and the amplitude of 1.9 mm. The measurements of the oscillation frequency and amplitude have been described elsewhere (Ref. 9). The welding speed, current and voltage were 7.6 mm/s (18 ipm), 100 A and 11 V, respectively.

The test welds were made coaxially with the prewelds, which were made wider so that, in the region they overlapped, the former could be contained within the latter. Two types of prewelds were made: a single-pass type and a multipass type. The single-pass type prewelds were intended to provide a dendritic structure upon which the test welds could be made. The welding speed, current and voltage were 5.9 mm/s (14 ipm), 100 A and 11 V, respectively. No arc oscillation was used. The multipass prewelds, on the other hand, were intended mainly to dissolve the heterogeneous nuclei in the area where the test welds were to be made. The welding speed, current and voltage were 0.64 mm/s (1.5 ipm), 45 A and 11 V, respectively. Ten consecutive passes were found sufficient for complete dissolution of heterogeneous nuclei.

The specimens were sectioned, polished and etched after welding, with a solution containing 75 ml HCl, 25 ml HNO₃ and 5 ml HF to reveal the grain structure. The specimens for scanning electron microscopy were electropolished using a solution of 200 ml phosphoric acid, 150 ml sulfuric acid, 275 ml water and 25 grams chromic oxide (Ref. 10). A JEOL JSM-35C scanning electron microscope was used for microstructural examination at high magnifications. An energy dispersive spectrometer (EDS) was used for the identification of particles within the grains. The beam size was around 200 Angstroms, and the volume of the specimens affected by the beam was on the order of one cubic micron.

Results and Discussion

Quenched Microstructure of the Welding Zone

Figure 1 shows the microstructure of the welding zone quenched during the GTA welding of 2219 aluminum alloy. Figure 1A shows the locations where the micrographs in Figs. 1B and C were taken. Figure 1B shows the microstructure along the leading portion of the pool boundary. As indicated by the brackets in the micrograph, the grains near the pool boundary were much smaller than those in the base metal, thus indicating partial melting of grains. In fact, the closer to the pool boundary, the smaller the grain size and the volume fraction of solid. The very fine solidification structure near the top of the photo is the liquid metal in the weld pool which solidified upon quenching.

Figure 1C shows the microstructure along the trailing portion of the pool boundary. The brackets in the micrograph indicate the region of growing dendrites. Partially melting grains and growing dendrites are the characteristics of the melting and solidification of alloys during welding, respectively. The higher the alloying content, or more appropriately, the wider the solidification temperature range, the more clear the partially melting grains and the growing dendrites are

Figure 2A shows the microstructure of a small portion of the weld pool metal ahead of the trailing portion of the pool boundary, i.e., position "Z" in Fig. 1A. As shown, a heterogeneous nucleus is evident at the origin of the equiaxed dendrite, which nucleated from upon the nucleus and grew in the weld pool during quenching. This heterogeneous nucleus is shown at higher magnifications in Figs. 2B and 2C. The energy dispersive spectrometer analysis of the nucleus is shown in Fig. 2D. As shown, the nucleus is rich in titanium, just like heterogeneous nuclei in aluminum castings and ingots (Al₃Ti or its similar compounds). The microcavities inside and along the grain boundary were originally occupied by the eutectic phase, which dissolved during etching. Similar results were observed in 2219 aluminum welds. It should be pointed out that this is the first time that heterogeneous nuclei in the weld pool were observed.

From the above discussion, the microstructure of the welding zone of an alloy can be shown schematically in Fig. 3. As shown, it consists of partially melting grains along the leading portion of the pool boundary, growing dendrites along the trailing portion of the pool boundary and, in some alloys, heterogeneous nuclei in the weld pool.

Mechanisms of Weld Metal Nucleation

Based on the microstructure observed, three different mechanisms of weld metal nucleation and grain refining can be introduced, as shown in Fig. 3. In order to help discuss these mechanisms, weld pool convection will be briefly described first.

As demonstrated recently by Kou, et al. (Refs. 11, 12), convection exists in the weld pool due to various driving forces,

such as the buoyancy force, the electromagnetic force and the surface tension gradient at the pool surface. Figure 4, for instance, shows the convection pattern in a GTA weld pool in a 3.2-mm (1/8-in.) thick 6061 aluminum sheet, due to the electromagnetic force. The calculated convection pattern is in the plane perpendicular to the welding direction and passing through the heat source. The velocity magnitude is indicated by the length of the arrows. The decrease in the velocity of the liquid metal towards the pool boundary suggests the existence of negative velocity gradients and, therefore, shear forces at the pool boundary. The purpose of Fig. 4 is to illustrate that considerable convection can exist in the weld pool. It is not implied that weld pool convection in a thinner workpiece, e.g., 1.6 mm (1/15 in.) thick, should be identi-

Dendrite Fragmentation

Weld pool convection can in principle cause fragmentation of the tips of the dendrites in the mushy zone, as illustrated in Fig. 3. These dendrite fragments are carried into the bulk weld pool, where, if able to survive the weld pool temperature, they can act as nuclei for new grains to form. It is interesting to note that this mechanism has been quoted very frequently as the grain refining mechanism for weld metals, even though there has been no proof at all.

Grain Detachment

In the case where partially melting grains are loosely held together by the liquid films between them, weld pool convection can make them detach themselves from the pool boundary and carry them into the weld pool, as also illustrated in Fig. 3. Like dendrite fragments, these partially melting grains, if they survive in the weld pool, can act as nuclei for the formation of new grains in the weld metal. Pearce, et al. (Ref. 13), have recently suggested that this is the mechanism responsible for grain refining in GTA welds of 7004 aluminum alloy made with weld pool stirring.

Heterogeneous Nucleation

According to the nucleation theory, atoms in a liquid metal have to overcome a critical energy barrier (ΔG^*) so that they can form solid nuclei that are larger than a critical size (r^*), in order to be stable and able to grow into solid grains (Ref. 14). Unfortunately, this critical energy barrier usually is high and difficult to overcome, if these nuclei are to be formed solely by the atoms in the liquid themselves, *i.e.*, the so-called homogeneous nuclei.

For this reason, constitutional super-

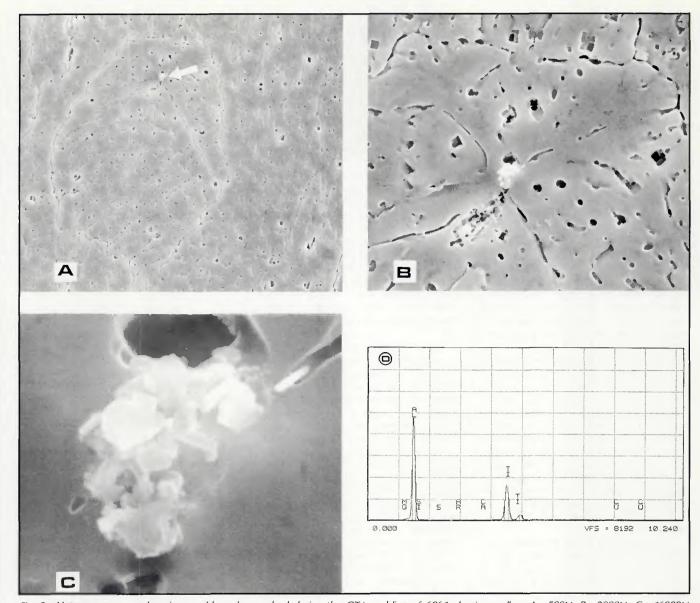


Fig. 2—Heterogeneous nucleus in a weld pool quenched during the GTA welding of 6061 aluminum alloy. A=500X; B=2000X; C=16000X; D=EDS analysis

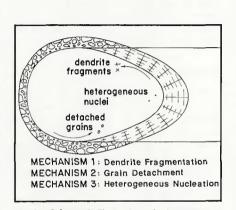


Fig. 3 – Schematic illustration of microstructure of the welding zone and nucleation mechanisms for the weld metal of alloys

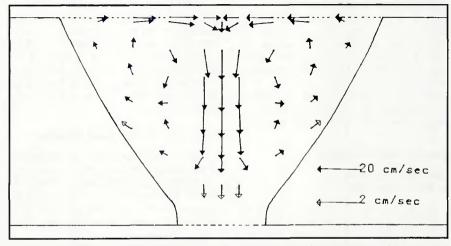


Fig. 4—Calculated pattern for the electromagnetic-force-induced convection in the transverse cross-section of a weld pool (front view) in 3.2-mm thick 6061 aluminum alloy. The velocity magnitude is indicated by the length of the arrows. From Kou, et al. (Ref. 13)

cooling is not expected to be significant enough by itself to cause nucleation under normal welding conditions, unless the alloys and the solute levels are such that exceptionally high constitutional supercooling can exist. As pointed out by Savage (Ref. 8), the formation of equiaxed grains due to constitutional supercooling alone requires such an extensive constitutional supercooling that it is not encountered in normal fusion welds. This is because the thermal conditions in the liquid pool are so much more severe during arc welding than during ingot casting that temperature gradients are too high to allow large enough constitutional supercooling for nucleation of equiaxed grains in the weld metal. However, if the liquid metal through inoculation contains a significant number of solid particles on which the atoms in the constitutionally supercooled liquid metal can be easily arranged in a crystalline form, it is no longer necessary for the atoms in the liquid metal to form nuclei solely by themselves. These particles, i.e., heterogeneous nuclei, are also shown in Fig. 3. Figure 5 illustrates the heterogeneous nucleation and subsequent growth of new grains during welding. Grain refining has been produced in welds of aluminum alloys (Refs. 10, 15), steels (Refs. 2, 4, 16) and titanium alloys (Ref. 17) by inoculation.

A Method for Identifying Nucleation Mechanisms

The mechanism of grain detachment is the easiest one to identify and, therefore, is suggested as the first candidate for identification. As shown in Step 1 of Fig. 6, overlap welding is carried out by first making a wide single-pass preweld exhibiting columnar grains near the fusion boundaries. This should be verified metallographically. Next, the test weld is made along the centerline of the preweld, such that its fusion boundaries are contained in the columnar grain regions of the preweld in the region of overlapping. A similar technique, in fact, has been used recently by Pearce, et al. (Ref. 13). As shown in the figure, if the equiaxed grains in the test weld disappear after it enters the overlap region, then grain detachment is the nucleation mechanism. This can be explained with the microstructure around the weld pool shown schematically in Fig. 7. As shown, the partially melting grains which act as the nuclei in the test weld are no longer available once it enters the overlap region, since the leading portion of the weld pool boundary is now surrounded by the dendritic structure of the preweld. If, on the other hand, the equiaxed grains in the test weld continue to exist after it enters the overlap region, then either dendrite fragmentation or heteroge-

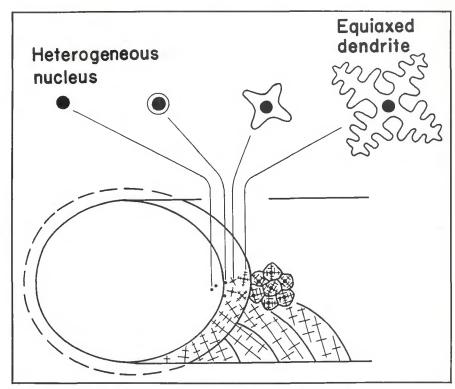


Fig. 5-Schematic illustration for the formation of equiaxed grains in the weld metal due to heterogeneous nucleation

neous nucleation can be the mechanism.

In order to find out which of the remaining two nucleation mechanisms is operating, overlap welding is again carried out in Step 2 for alloys in which heterogeneous nuclei, if they exist, can be dissolved. For instance, Al₃Ti nuclei in aluminum containing less than 0.12 wt-% Ti, as can be seen from the Al-Ti phase diagram shown in Fig. 8, can be dissolved by heating to around or above the peritectic temperature, i.e., 665°C (1229°F). Such Al₃Ti nuclei or their equivalent forms usually come from the titanium-rich master alloys (up to 5 wt-% Ti) used for casting. The master alloys are added to the liquid metal of proper composition and the liquid metal is poured before these nuclei dissolve completely, in order to achieve grain refining in the castings or ingots. In particular, the holding temperature of the liquid metal should not be too high and the holding time should not be too long, if complete dissolution of the nuclei is to be avoided (Refs. 19, 20). The preweld in Step 2 is a multipass weld intended for the dissolution of such nuclei. As will be shown later, the dissolution of nuclei is verified by SEM. For materials with a high Ti level, e.g., higher than the peritectic composition, Ti-rich nuclei, after being dissolved, reform during solidification, and this method is not recommended for testing the heterogeneous nucleation mechanism.

If the equiaxed grains in the test weld

continue to exist after it enters the overlap region, then dendrite fragmentation can be the nucleation mechanism. This again can be explained with the microstructure around the weld pool shown schematically in Fig. 7. As shown, dendrite fragmentation can occur both before and after the test weld enters the overlap region, thus producing equiaxed grains in the entire test weld. If, on the other hand, the equiaxed grains in the test weld disappear after it enters the overlap region, then heterogeneous nucleation is the mechanism. This is because the heterogeneous nuclei are no longer available once the test weld enters the overlap region, as illustrated in Fig. 7. For alloys with heterogeneous nuclei which are difficult to dissolve or tend to reform after being dissolved, Step 2 can be skipped.

Perhaps the most direct way to check whether the equiaxed grains have nucleated heterogeneously is to examine them under the scanning electron microscope, particularly the area near the origin of the dendrites. If heterogeneous nucleation is the mechanism, then heterogeneous nuclei can show up at the dendrite origin, as shown schematically in Step 3 of Fig. 7. The energy dispersive spectrometer can be used for analyzing qualitatively the composition of the nuclei. It is worth mentioning that some of the equiaxed grains which do not seem to have heterogeneous nuclei actually have them below the polished surface of the speci-

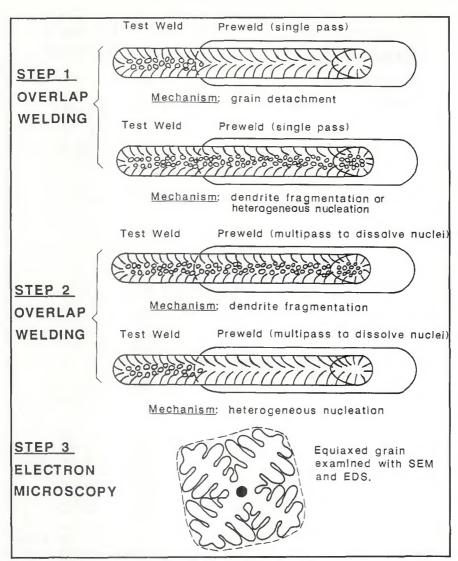


Fig. 6 – A step-by-step method for identifying the nucleation mechanism

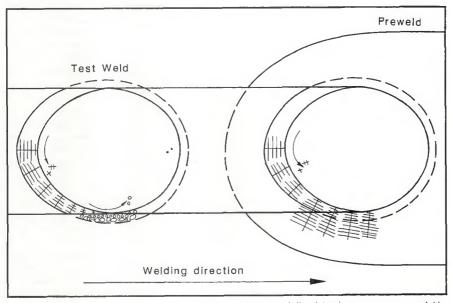


Fig. 7 — Changing the microstructure around the weld pool and dissolving heterogeneous nuclei by overlap welding

men, or already have lost them during polishing. Further polishing, usually electrochemically, can help reveal the nuclei below the surface of the specimen. In some alloys (e.g., 5052 aluminum), the coarsening effect during solidification is so severe that dendrite arms are fused or sintered together and the dendritic structure becomes rather ill defined, thus making the origins of the dendrites rather difficult, if not impossible, to locate. For these alloys, and possibly alloys in which heterogeneous nuclei are too tiny to detect, the overlap welding suggested in Step 2 is more useful.

Identification of Nucleation Mechanisms in Aluminum Welds

The procedure suggested above was applied to identify the grain refining mechanism in GTA welds of commercial 6061 aluminum alloy made with circular arc oscillation at 35 Hz.

Since arc oscillation is expected to promote weld pool convection, both grain detachment and dendrite fragmentation are likely to be operating. In order to test the mechanism of grain detachment, overlap welding was carried out using single-pass prewelds. Figures 9A and 9B show the grain structure of the test weld outside and inside the overlap region, respectively. As shown, equiaxed grains existed in the test weld both outside and inside the overlap region, thus confirming that grain detachment was not the mechanism responsible for grain refining.

In order to find out whether dendrite fragmentation or heterogeneous nucleation was responsible for grain refining, overlap welding was carried out using multipass prewelds. Figure 10A shows the grain structure of the test weld in the region where it overlapped with a preweld made with six melting passes. By comparing it with the micrograph shown in Fig. 9A, it can be seen that the number of equiaxed grains decreased. As shown in Fig. 10B, the test weld in the overlap region was essentially free of equiaxed grains, when the preweld was made with ten melting passes. This suggested that heterogeneous nucleation was responsible for grain refining in the oscillated arc weld of 6061 aluminum. This finding is surprising from the viewpoint that commercial 6061 aluminum is not known to contain titanium, at least not according to the specifications of the alloy (Ref. 21). Detailed chemical analysis, shown in Table 1, did indicate a titanium level of 0.043 wt-%, however. It should be pointed out that chemical analysis on 6061 aluminum from a different supplier also showed similar results. In fact, similar and somewhat lower titanium levels were respectively observed in commercial 5052 and 2014 aluminum alloys (Table 1), even though they are not known to contain titanium either. Although no multi-component phase diagrams are available for 6061 aluminum alloy, the titanium level of 0.043 wt-% in this material was perhaps too low to allow the titanium-containing heterogeneous nuclei, e.g., Al₃Ti, to reform after being dissolved.

It is worth mentioning that the columnar grains in the preweld became coarser after multipass melting. This, however, should not affect the conclusion that heterogeneous nucleation, rather than dendrite fragmentation, is the mechanism of nucleation. This is because dendrite fragmentation is related to the dendritic (subgrain) structure, rather than the grain structure. Also, since the welding condition for the test weld remained unchanged, the growth velocity of epitaxial columnar grains from the fusion boundaries of the test weld also remained unchanged. In other words, the decrease in the number of fine grains in Fig. 10 is not the result of enhanced epitaxial growth due to multipass melting.

In order to further confirm that the equiaxed grains in the oscillated arc weld of 6061 aluminum nucleated heterogeneously, the test weld was examined under a scanning electron microscope. Figure 11 shows the heterogeneous nucleus in a small grain in the test weld. As shown, the grain had nucleated heterogeneously from the particle at the origin of the dendrite. The nucleus is rich in titanium, as indicated by the EDS analysis shown in Fig. 11D. It is worth noting that the origin of the dendrite is not located at the geometric center of the grain, due to the fact that the local temperature gradient was not the same in all directions and that the dendrite grew preferentially in

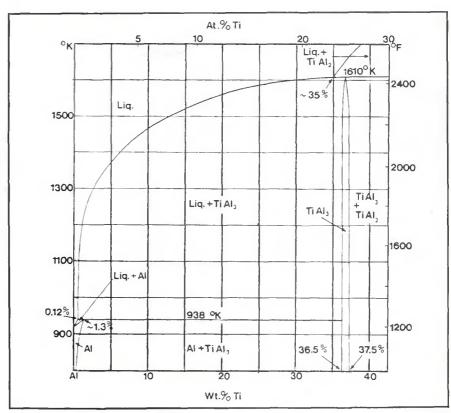
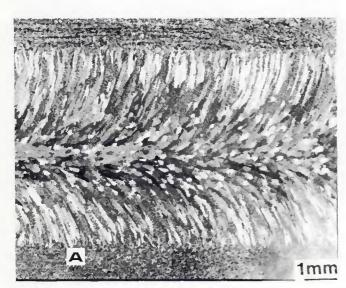


Fig. 8 – The aluminum-rich side of the aluminum-titanium phase diagram. From Mondolfo (Ref. 18)

Table 1—Chemical Analysis of Aluminum Alloys Welded (wt-%)											
	Mg	Si	Cu	Fe	Mn	Cr	Ni	Zn	Ti	Zr	Al
6061	1.2	0.6	0.2	0.4	0.07	0.2	0.01	0.09	0.04	0.00	balance
5052	2.2	0.1	0.03	0.3	0.08	0.2	0.01	0.02	0.04	0.00	balance
2014	0.5	8.0	4.3	0.3	8.0	0.03	0.00	0.06	0.01	0.00	balance



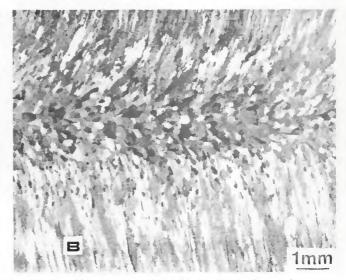


Fig. 9 — Grain structure of an oscillated arc weld of 6061 aluminum which overlapped with a single-pass preweld. A — outside the overlap region; B — inside the overlap region

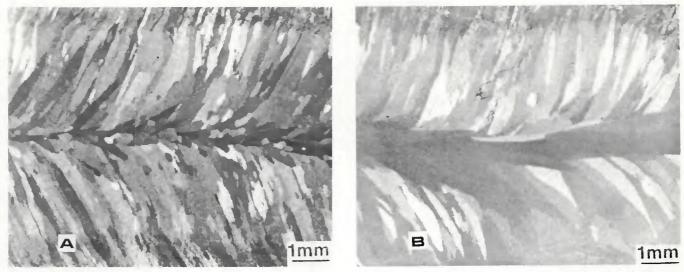


Fig. 10 – Grain structure of oscillated arc welds of 6061 aluminum in the region of overlapping. A – a preweld made with six melting passes; B – a preweld made with ten melting passes

the direction of the maximum temperature gradient. It is also worth noting that the clear evidence of heterogeneous nuclei in this study confirms the recent studies of Kerr, et al. (Refs. 10, 13), in which the evidence of heterogeneous nuclei was shown but was not as clear.

The results described above are consistent with the heterogeneous nuclei found in the weld pool by quenching, e.g., Fig. 2. However, it should be pointed out that the existence of heterogeneous nuclei in the weld pool is only a necessary, but not sufficient, condition

for grain refining of the weld metal. This and the effect of welding parameters on weld metal nucleation and grain refining will be addressed in subsequent reports.

Figure 12A shows the heterogeneous nucleus in a grain in the overlap region between the test weld and a preweld

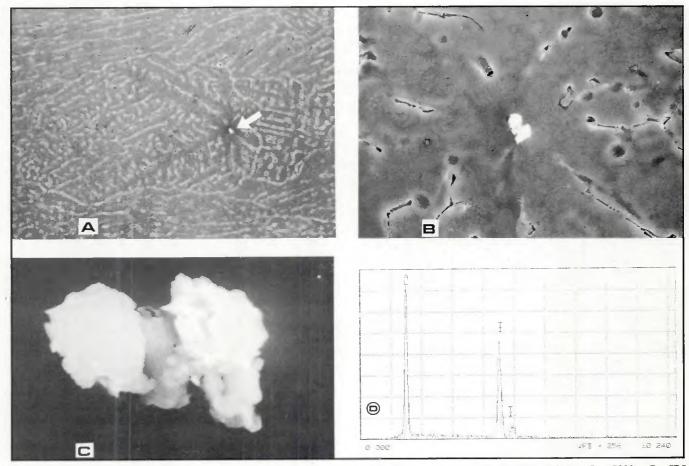


Fig. 11—Heterogeneous nucleus in a small grain in an oscillated arc weld of 6061 aluminum alloy. A = 330X; B = 2000X; C = 15000X; D = EDS analysis

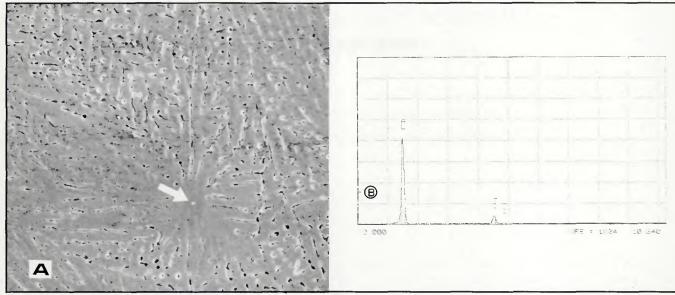


Fig. 12 — Heterogeneous nucleus in a grain in the overlap region between an oscillated arc weld of 6061 aluminum and a preweld made with six melting passes. A = 330X; B = EDS analysis

made with six melting passes. It is noteworthy that this nucleus and other nuclei observed in the same weld are considerably smaller than that shown previously in Fig. 11, due to dissolution during the multiple melting passes of the preweld. Also, according to the EDS analysis shown in Fig. 12B, the titanium content of this nucleus is significantly lower than that of the nucleus shown previously in Fig. 11. This is because the smaller the nucleus, the more the EDS analysis reflected the composition of the aluminum-rich background material, even if there is no difference in the composition of the nuclei.

Conclusions

1. The microstructure of the welding zone during welding, in particular heterogeneous nuclei, partially melting grains and growing dendrites, has been clearly shown in this study. It is an important key to the understanding of weld metal nucleation and grain refining.

2. Heterogeneous nuclei in the weld pool have been observed for the first time. The nuclei in the weld pools of 6061 and 2219 aluminum alloys are rich in titanium.

3. The mechanisms of dendrite fragmentation, grain detachment and heterogeneous nucleation are well explained by the microstructure of the welding zone.

4. A method for identifying the nucleation mechanism of the weld metal of alloys has been demonstrated. This has been made possible by changing the microstructure around the weld pool and dissolving heterogeneous nuclei through overlap welding, and by examining the grains of the weld metal with electron

microscopy.

5. As an illustration of this method, the nucleation mechanism responsible for grain refining in an oscillated arc weld of commercial 6061 aluminum alloy has been identified as heterogeneous nucleation, rather than dendrite fragmentation or grain detachment.

6. The heterogeneous nuclei observed in the weld metal of 6061 aluminum alloy are rich in titanium, even though this alloy is not known to contain titanium. Detailed chemical analysis, which indicates a low but significant titanium level of 0.043 wt-% in this alloy, confirms the presence of these titanium rich nuclei.

The present study is significant in that it clearly reveals the microstructure in the welding zone during welding and ties it with nucleation and grain refining mechanisms, so that the important subject of weld metal grain refining can be better understood. Furthermore, a method is provided for identifying the right mechanism responsible for nucleation and grain refining of the weld metal.

Acknowledgments

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