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Microsegregation in Partially Melted Regions of 70Cu-30Ni Weldments

Evidence supports an hypothesis that explains the mechanism of partial melting and its effects

BY W. F. SAVAGE, E. F. NIPPES AND T. W. MILLER

ABSTRACT. The partially melted regions of gas tungsten-arc welds made in a 70Cu-30Ni alloy were studied. An hypothesis explaining the mechanism of partial melting was advanced and tested by employing point-count electron beam microprobe analysis of the microstructure of the partially melted regions of welds made in this material. The analysis showed that segregation (probably associated with the worked cast ingot structure) was present in the base metal and therefore resulted in localities with lower effective melting temperature. The localities with lower effective melting temperatures adjacent to the fusion zone of the welds melted, while localities with higher effective melting temperatures did not melt and thus partial melting occurred. Metallographic evidence was presented in support of the hypothesis showing that grain growth in the base metal which occurred adjacent to the fusion zone during welding was inhibited by the presence of the melted localities. Furthermore, the extent of the partially melted region was influenced by segregation bands present in the base metal.

W. F. SAVAGE is Professor of Metallurgical Engineering and Director of Welding Research and E. F. NIPPES is Professor of Metallurgical Engineering, Rensselaer Polytechnic Institute, Troy, New York 12181. T. W. MILLER, formerly INCRA Research Fellow at R.P.I., is now Research Engineer in Steel Making, Homer Research Laboratory, Bethlehem Steel Corp., Bethlehem, Pa.

Introduction

Most commercially processed alloys exhibit solute segregation which results from the solidification of the original ingot. Metallographic evidence of such segregation is present as coring in castings and as banding and stringers of inclusions in wrought products.

There are two distinct regions of melting present in autogenous welds. The first region is the fusion zone in which there is 100% melting. The fusion zone is therefore bounded by the locus of the effective liquidus temperature of the weld material (Ref. 1). The second region, which was of interest in the present investigation, is the partially melted region. This region lies between the fusion zone and the unmelted base metal and is bounded by the locus of the liquidus temperature on one side (the fusion line) and by the locus of the effective solidus on the other side (the beginning of the true heat-affected zone).

Owczarski, Duvall and Sullivan (Ref. 2) showed that a partially melted region in the heat-affected zone adjacent to the fusion line of welds in each of two nickel base superalloys was the most crack sensitive region of the base metal in the vicinity of the weld.

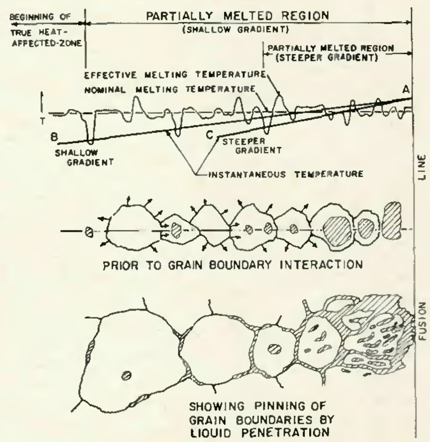


Fig. 1 — Proposed mechanism for partial melting in a single phase alloy

They proposed a model relating grain boundary liquation — which initiated in the proximity of MC type carbides during rapid heating — to hot ductility properties and heat-affected zone cracking.

An Hypothesis to Explain Partial Melting

In the presence of point-to-point variations in solute content in the base metal, the effective melting temperature would vary from place to place. A schematic representation of the variation in the effective melting temperature caused by residual

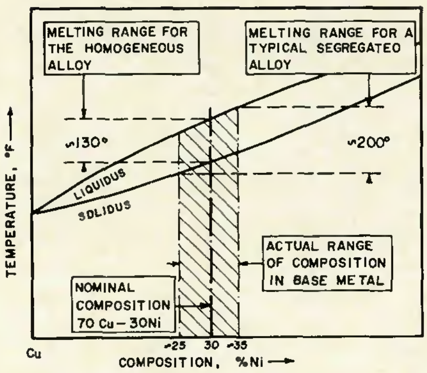


Fig. 2 — The influence of segregation on the melting range of a binary alloy

Table 1 — Composition of 70Cu-30Ni Alloy Anaconda American Brass Heat No. 26881[a]

Element	Weight percent
Copper	68.96
Nickel	29.62 (by difference)
Zinc	<0.10 (<0.005 [b])
Iron	0.52
Manganese	0.87
Phosphorous	0.008
Lead	<0.005 (<0.02 [b])
Carbon	0.019
Bismuth	<0.003

(a) Analysis by Anaconda American Brass. No data on fabrication were available, other than that the material was finished by cold rolling to 3 sizes: 1/2, 1/8 and 1/16 in. thicknesses.
(b) Electron beam microprobe analysis supplied by Advanced Metals Research Corp.

segregation in the base metal is presented at the top of Fig. 1.

As defined above, the fusion zone of the weld is that region which experiences peak temperatures above the liquidus and is characterized by complete melting. Adjacent to this region, a volume of base metal experiencing peak temperatures ranging downward from the effective liquidus to the effective solidus must experience partial melting. The mechanism by means of which partial melting occurs is not understood and therefore the following hypothesis is advanced to explain this phenomenon.

Two typical temperature gradients have been super-imposed on the schematic showing the point-to-point variation in the effective melting temperature at the top of Fig. 1. Note that the localities where the effective melting temperature is below the instantaneous temperature will experience localized melting. These melted areas are shown schematically in the center of Fig. 1 for the shallow temperature gradient, (line AB). (It is assumed that no movement of the grain boundaries has yet occurred at this point in the discussion).

It has been well established that the regions adjacent to the fusion zone are exposed to a high peak temperature for a finite period of time. This exposure causes grain-boundary migration to occur and in the absence of any second phase would result in normal grain growth. However, the melted regions present act as a second phase to retard the motion of these migrating boundaries. In fact, the liquid phase, being similar in composition, should wet the boundaries readily causing pinning of the boundaries and arresting the normal grain growth process. This grain-boundary-pinning phenomenon is summarized in Fig. 1 (center and bottom).

The extent of melting present and the separation between localized molten regions determine the distance over which grain boundaries can migrate before becoming pinned and thus determine the maximum grain size. Figure 1 (bottom) shows that grain growth is limited wherever the melted (shaded) areas are present. Note that both the fraction melted and the spacing between molten localities should have a strong influence on the resultant grain size in the partially melted region. Specifically, either increasing the fraction melted, or decreasing the distance between melted localities, decreases the distance a grain boundary can migrate before being pinned. Thus, the maximum grain size should result at the left in Fig. 1 where the extent of partial melting approaches zero (defined as the edge of the true heat-affected zone) and the grain size should decrease as the fusion line is approached. This is in contrast to the behavior expected under conditions leading to normal grain growth where the maximum grain size would increase continuously as the temperature of exposure is increased.

The width of the partially melted region depends upon both the temperature gradient produced adjacent to the weld and the magnitude of the peak-to-peak variations in solute concentration in the original base metal.

The influence of the peak-to-peak variation in solute concentration can be seen by reference to Fig. 2 (Ref. 5). For a homogeneous 70Cu-30Ni alloy the solidus and liquidus temperature correspond to 2130 F (1166 C) and 2260 F, (1238 C), respectively. Thus the partially melted region for the perfectly homogeneous alloy would extend over a 130 F (72 C) interval of the temperature gradient beyond the locus of the liquidus, i.e., the fusion line.

On the other hand if one assumes a ±5% variation in nickel content due to residual segregation in the base material, the nickel rich regions would exhibit a liquidus of 2100 F (1149 C) while the nickel depleted regions would exhibit a solidus of 2300 F (1260 C). Thus, the partially melted region would extend over a 200 F (111 C) interval of the temperature gradient beyond the locus of the liquidus.

Referring again to Fig. 1, note that with the steeper temperature gradient, (line AC), the extent of partial melting would be greatly reduced compared to the extent of partial melting with the shallow gradient (line AB) for the same conditions of residual segregation in the base material. Furthermore, according to the model, smaller peak-to-peak variations in solute content in the original base material would reduce the point-to-point variations in the effective melting temperature and thus restrict the extent of partial melting with a given temperature gradient.

Object

The objectives of this investigation were:

1. To test an hypothesis explaining the mechanism by which partial melting occurs in welds made in a 70 copper-30 nickel alloy.
2. To determine the extent of solute segregation present in the partially melted region of a weld made in a 70 copper-30 nickel alloy.
3. To investigate the effects of welding parameters on the partially melted region of welds made in a 70 copper-30 nickel alloy.

Materials and Procedure

Exploratory Welds

A series of autogenous gas tungsten-arc (GTA) exploratory welds were made with a wide range of welding parameters on cold rolled 70 copper-30 nickel of 1/8 in. and 1/16 in. thickness rolled from Heat Number 26881. The analysis of this heat is summarized in Table 1. Unfortunately, no processing data are available, other than that the material was finished to size by cold rolling.

Table 2 summarizes the welding conditions for the series of exploratory GTA welds made for this investigation. Pure argon was employed as a shielding gas for all exploratory welds. The actual welding was performed in a sealed atmosphere chamber maintained under a slight positive pressure of pure argon by a controlled leak. The primary source of gas during welding was the electrode holder, while during standby a low flow rate of argon was supplied from a separately regulated source to maintain the positive pressure differential.

Duplicate welds approximately 4.5

Table 2 — Welding Conditions and Identification Code for Exploratory Welds Made on 70Cu-30Ni Alloy

Electrode	— EWTh-2, centerless ground, 1/8 in. diam
Electrode tip	— Conical, ground to 90 deg included angle
Electrode ext.	— 1.75 in. from collet
Arc length	— 0.060 in. from cold workpiece
Arc voltage	— 8.5 ± 0.5 V
Torch gas	— 35 cfh argon (in argon-filled sealed-atmosphere chamber)
Weld geometry	— See text

Current	50 A	100 A		150 A		200 A	
Code	A	B		C		D	
Thickness (in.)	1/16	1/16	1/8	1/16	1/8	1/16	1/8
Code	a	a	b	a	b	a	b
Travel speed							
5	Aa5 [(a)]	Ba5	Bb5	X [(b)]	Cb5	X	Db5
10	Aa10	Ba10	Bb10	Ca10	Cb10	X	Db10
20	— [(c)]	Ba20	—	Ca20	Cb20	Da20	Db20
40	—	Ba40	—	Ca40	Cb40	Da40 [(d)]	Db40

(a) Aa5, atc. speciman codes for welds made
(b) X indicates complete melt-through
(c) — indicates no weld bead was producad
(d) specimen Da40 was used for electron beam microprobe analysis

Table 3 — Welding conditions of Varestraint Specimen V-2 Used for Electron Beam Microprobe Analysis

Electrode	— EWTh-2, centerless ground, 1/8 in. diam
Electrode tip	— Conical, ground to 90 deg included angle
Electrode ext.	— 1.75 in. from collet
Arc length	— 3/16 in. from cold workpiece
Arc voltage	— 10 ± 0.5 V
Torch gas	— 35 cfh argon
Specimen size	— 2 × 12 × 0.46 in.
Weld current	— 200 A
Travel speed	— 2.5 ipm
Augmented strain	— 4 percent

Table 4 — Etching Reagent Employed for Metallography of Electron Beam Microprobe Analysis

Stock Solution

Quantity	Component
1900 ml	H_2O
40 gr	CrO_3
15 ml	HNO_3 conc.
50 ml	H_2SO_4 conc.
7.5 gr	NH_4Cl

Directions

Dilute with alcohol, 1 part stock to 3 parts alcohol (C_2H_5OH).
Etch by immersion, agitate.
Use immediately, very short lived.

in. long were suitably spaced on 1.5 × 5 in. specimens using the conditions summarized in Table 2. The specimens were clamped on a massive 3 in. thick water cooled aluminum platen using 1/8 in. thick steel shims to elevate the undersurface of the specimen above the platen. Copper hold-down bars approximately 1/4 × 1 × 5 in., located parallel to and equidistant from the longitudinal centerline of the weld, were employed both to restrain the weld from buckling and to provide an additional heat sink.

These welds were photographed in the as-welded condition at 7X to document their surface macrostructure. The welds were then sectioned in three mutually perpendicular directions, prepared metallographically, and photographed at 50X. The metallographic sections were taken: (1) transverse to the welding direction, (2) parallel to the top surface near the as-welded surface, and (3) perpendicular to the top surface and parallel to the welding direction at the centerline of the weld.

Modified Marble's reagent was used as an etchant. By this procedure both the macrostructures and the microstructures of the welds were documented. One of these specimens (Da 40, transverse) was prepared for electron beam microprobe analysis.

Varestraint Testing

The 70Cu-30Ni material was welded and strained in the R.P.I. Varestraint apparatus (Ref. 6,7). Specimens were machined from nominally 1/2 in. cold rolled stock of Heat #26881 to a final dimension of 2 × 12 in. One 2 × 12 in. surface was machined to provide a suitable welding surface and the resulting specimen thickness was held to 0.46 ± 0.01 in. Following cleaning with acetone and wiping dry, the specimens were positioned in the Varestraint apparatus. Autogenous GTA welds were then made on the specimens, and the specimens were subjected to a 4% augmented strain, the greatest augmented strain possible with existing equipment. Table 3 summarizes the test procedure and welding conditions employed.

The specimens were sectioned metallographically both parallel to the surface of the weld and along a plane perpendicular to the surface and transverse to the welding direction. The resulting samples were polished and etched with the etchant described in Table 4. One of these specimens (V-2, parallel to the surface) was subsequently prepared for electron beam microprobe analysis.

Electron Beam Microprobe Analysis

Two specimens were prepared for electron beam microprobe analysis: Specimen Da 40 (a transverse section of an exploratory weld) and Specimen V-2 (sectioned parallel to the surface of a Varestraint specimen which was subjected to 4% augmented strain).

The mounted specimens were carefully polished and etched with the etchant described in Table 4. Photomicrographs were taken at 100X of the as-polished condition and at both 100X and 500X of the etched specimens.

After suitable areas for microanalysis had been identified from the photomicrographs, the specimens were repolished with 0.05 micron Al_2O_3 and lightly re-etched. The areas of interest were then relocated using the photomicrographs as guides and identified by suitably located microhardness indentations.

The electron beam microprobe analysis was performed by Ad-

		% Ni	% Mn	% Fe	% P	% Zn
GRAIN BOUNDARIES	A	28.6	1.05	0.51	0.011	0.014
DOTS	B	29.0	0.94	0.51	0.011	0.034
GRAIN CENTERS	C	30.6	0.91	0.57	0.006	0.012

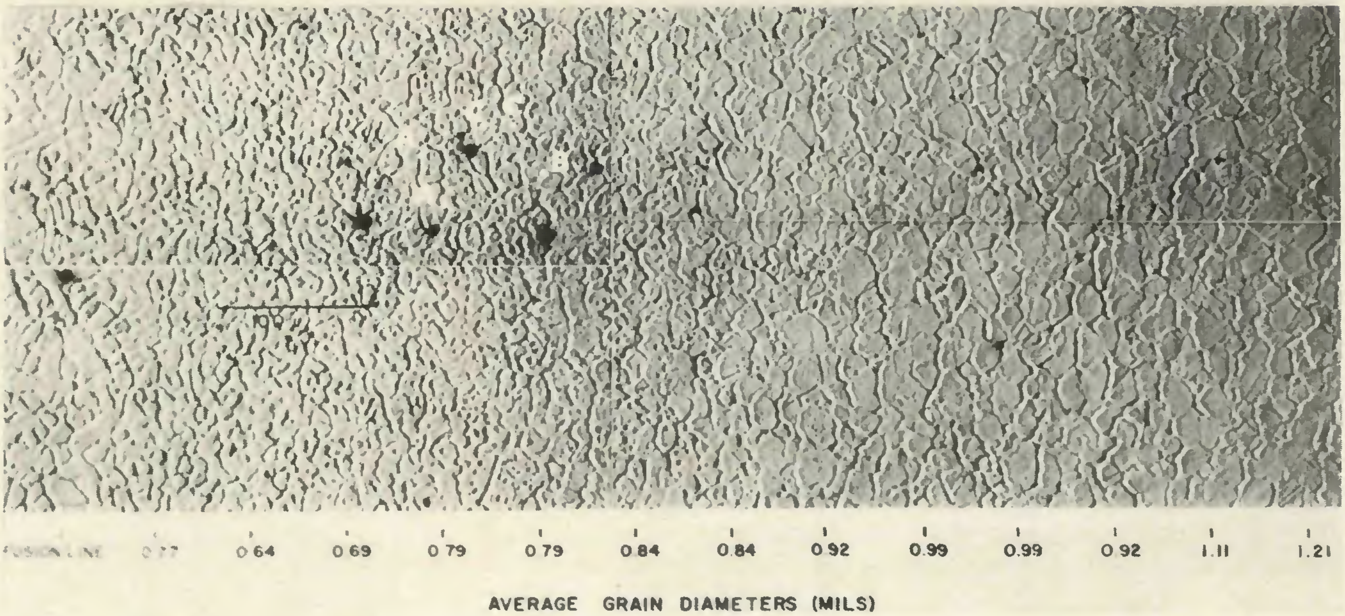


Fig. 3 — Partially melted region of specimen Da 40 near the root of the weld with average compositions at various features and average grain diameters

vanced Metals Research Corp. (AMR) at Burlington, Mass. The intensity of the back-scattered characteristic radiation excited by a 1 micron diameter stationary electron beam was measured by point-count techniques. It is estimated that this 1 micron diameter electron beam operating at 30 kV on a cupronickel alloy excites a 3 micron diameter hemisphere. With the aid of suitable standards and calibration curves available at AMR, the point-count data were converted to weight percent values for nickel, manganese, iron, zinc, phosphorous and lead.

Results and Discussion

Electron Microprobe Analysis Studies

The electron beam microprobe was employed to study segregation in the partially melted region of Specimen Da 40. Figure 3 shows a transverse section of the partially melted region at the root of the weld in Specimen Da 40. This specimen experienced almost full weld penetration and therefore exhibited a region where heat could be withdrawn only in two dimensions. This region thus experienced a shallow thermal gradient and, hence, a relatively large partially melted region resulted.

The electron beam microprobe analysis data tabulated in Fig. 3 show that the broadened grain boundaries and the "dots" in the partially melted region of Da 40 are slightly lower in nickel and iron than the grain centers. In addition, these boundaries and dots are higher in manganese, phosphorous and zinc. Thus all of the solutes are segregated in a manner which lowers the effective melting temperature.

Figure 4 shows a portion of the partially melted region of Specimen V-2 located near the as-welded surface. Since the weld in this specimen exhibited only partial penetration of the 1/2 in. thickness, the temperature gradient in the region of partial melting was relatively steep and the partially melted zone is less extensive than that of Da 40.

When the partially melted region of V-2 was subjected to electron beam microprobe analysis, spots T and T′ exhibited 36.4 and 37.2 percent nickel respectively, and thus had relatively high effective melting temperatures. Therefore these regions, although near the fusion line, did not melt.

By comparison regions Q, V, and W had compositions only slightly higher than the nominal composition of 29.6%, but apparently failed to melt because the peak temperature they experienced was below their effective melting temperature. On the other hand, spot S, with 32.0% nickel experienced melting because it was close to the fusion line where the peak temperature exceeded its effective melting temperature. Note that point S, with 32% nickel, melted while the adjacent point T, with 36.4% nickel failed to melt in spite of the fact that both points must have experienced almost the same peak temperature.

S′, S″, and S‴ are all localities of low nickel and all appear to have melted. Point S′ has a nickel concentration of 14.5 % and therefore much of the region surrounding it appears to have melted. The microanalysis data at this point are probably reasonably accurate since the region was wide enough to contain the entire excited volume. The progressively higher nickel contents obtained for points S″, S‴, P and O are probably less reliable since the 3 micron excited diameter approached or exceeded the width of the molten region (refer to Fig. 4). Thus it is likely that the true composition at these points is lower than the reported values for solutes with k greater than 1.0 (Ni and Fe) and higher than the reported values for solutes with k less than 1.0 (Mn, P, Zn and Pb).

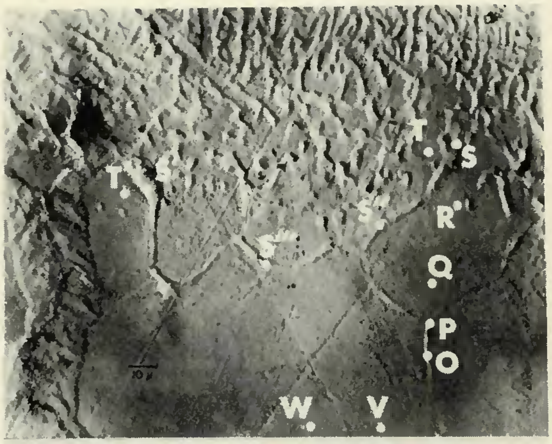
It will be recalled that k, the distribution coefficient, is defined as the ratio of the solute composition of the solid to the solute composition of the liquid for any temperature at which solid and liquid phases coexist (Refs. 1,8). If the solute tends to raise the melting temperature range, as is the case for Ni and Fe in copper base alloys, the liquid phase is depleted in solute and k has values greater than 1.0. On the other hand, if the solute, such as Mn, P, Zn and Pb in copper-base alloys, tends to lower the melting temperature range, the liquid phase is enriched in solute and k has a value less than 1.0.

Results of Grain Size Studies

Figures 3 and 5 show the partially melted regions at the roots of welds Da 40 and Aa 10, respectively. The average grain diameter, measured by the grain-boundary-line intersection technique, as a function of the distance from the fusion line is tabulated on both these figures. Note that in both cases there is an increase in the average grain diameter within the partially melted region at the fusion line as the distance from the fusion line increases. This behavior is consistent with the grain boundary pinning mechanism discussed previously in the hypothesis. Regions of extensive melting, located near the fusion line and experiencing extremely high temperatures, showed less grain boundary movement and thus exhibited a smaller grain size than regions more distant from the fusion line where the peak temperature was lower but less melting was present.

Specimen Aa 10 experienced 3-dimensional heat flow at the root of the weld and thus had a steeper thermal gradient than that which occurred in Da 40. Thus the temperature gradient in Specimen Aa 10 might correspond to the steeper peak temperature gradient (line AC in Fig. 1). On the other hand the temperature gradient in Specimen Da 40 would correspond to the shallow gradient represented by line AB in Fig. 1. Hence, the hypothesis explains the difference in sizes of the partially melted regions of Specimens Da 40 and Aa 10.

A similar correspondence between the steepness of the gradient and the width of the partially melted zone occurred in the other welds of the exploratory series. In the case of welds



COMPOSITIONS AT VARIOUS LOCATIONS IN PARTIALLY MELTED REGION IN VARESTRAINT SAMPLE V-2

	Ni	Mn	Fe	P	Zn	Pb
O GRAIN BOUNDARY	28.4	1.02	0.49	0.010	<0.005	<0.02
P GRAIN BOUNDARY	27.6	0.99	0.43	0.023	0.043	<0.02
O MATRIX	31.2	1.15	0.54	0.020	0.020	<0.02
R MATRIX	33.3	0.82	0.63	0.009	<0.005	<0.02
S GRAIN BOUNDARY	32.0	0.87	0.54	0.007	<0.005	0.06
S' GRAIN BOUNDARY	14.5	0.87	0.16	0.020	<0.005	<0.02
S'' GRAIN BOUNDARY	22.2	1.00	0.41	0.013	<0.005	<0.02
S''' GRAIN BOUNDARY	26.1	0.96	0.46	0.005	0.012	0.02
T MATRIX	36.4	0.91	0.76	0.004	0.049	0.02
T' MATRIX	37.2	0.89	0.73	0.001	0.022	0.04
V MATRIX	30.9	0.97	0.53	0.009	<0.005	0.04
W MATRIX	30.8	0.96	0.55	0.007	0.010	0.03

3μ EXCITED SPOTS ARE DRAWN TO SCALE IN PHOTO

Fig. 4 — Composition at various locations in partially melted region of Varestraint sample, V-2

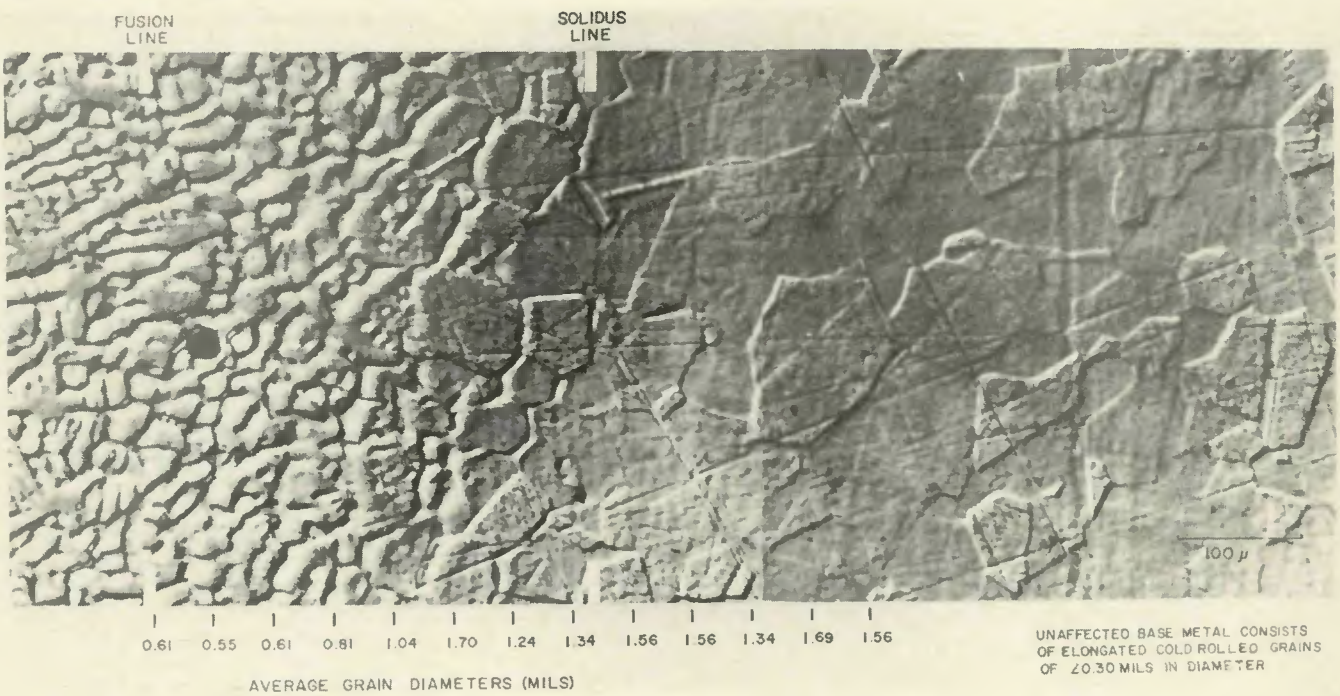


Fig. 5 — Partially melted region of specimen Aa 10 near the root of the weld showing average grain diameter as a function of location

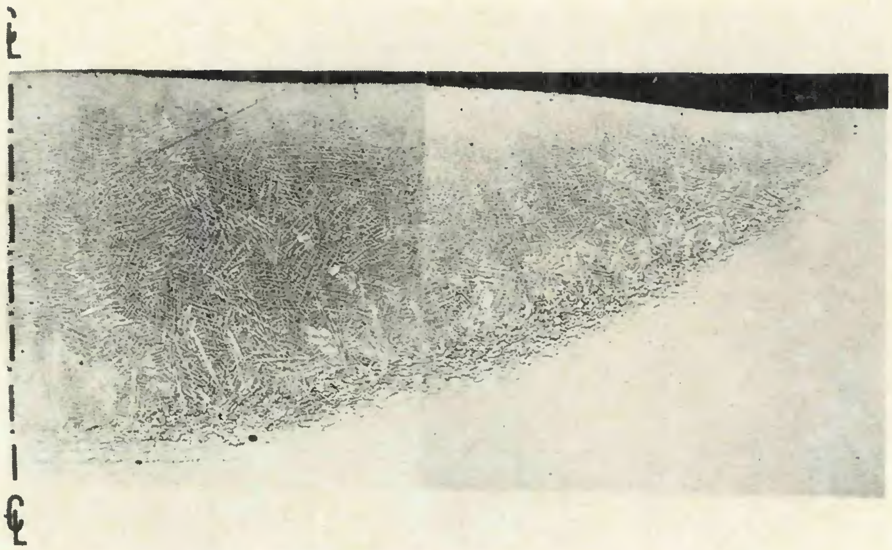


Fig. 6 — Photomicrograph of specimen Cb 10, transverse section. Weld made on 1/8 in. thick material. Etched with modified Merble's reagent. X50, reduced 20%

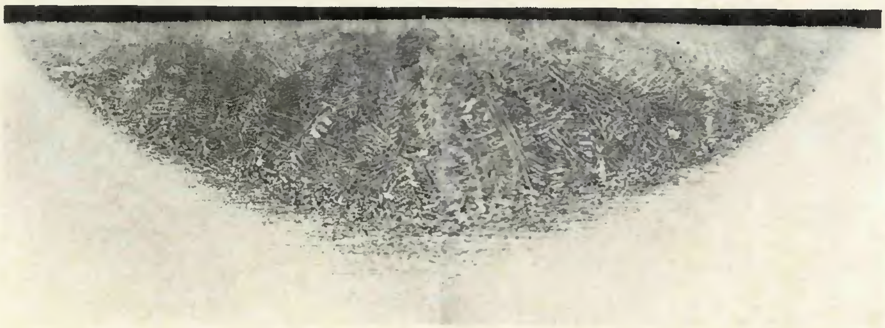


Fig. 7 — Photomicrograph of specimen Ba 20, transverse section. Weld made on 1/16 in. thick material. Etched with modified Marble's reagent. X50, reduced 20%

Fig. 8 — Photomicrograph of partially melted region of specimen V-2 near the top surface of the weld showing two large backfilled cracks. Etchant described in Table IV. X250, reduced 41%

having the same width and penetration, the welds made with fast travel speeds exhibited smaller partially melted regions than welds made with slow travel speeds. Since, for welds of the same size, taster travel speeds produce steeper thermal gradients adjacent to the weld fusion zone, the above observation is also consistent with the hypothesis.

Figures 6 and 7 show the partially melted regions of welds made in 1/8 in. and 1/16 in. thick material, Specimens Cb 10 and Ba 20, respectively. Both of these specimens exhibit striations in their as-received microstructure oriented parallel to the cold rolled surface. These striations are believed to be evidence of microsegregation in the original base metal before welding and are probably regions of alternately high and low solute content produced by rolling the cored ingot. Since the 1/16 in. thick material was cold reduced twice as much as the 1/8 in. material, the striations in the 1/16 in. material are more closely spaced than those of the 1/8 in. material. (Compare Figs. 6 and 7).

However, in both cases the partially melted regions of the welds show point-to-point variations in width, being wider where they intersect the striations. This tendency for the partially melted region to extend a greater distance in the vicinity of such striations supports the hypothesis regarding local variations In original base metal composition.

There appeared to be some metallographic correlation between the hot cracks produced adjacent to the fusion line of the Varestraint specimens and features in the partially melted region. Figure 8, (a portion of which was shown in Fig. 4) is a section near the as-welded surface of Specimen V-2, and shows two backfilled cracks in the partially melted region. It is probable that these cracks were nucleated at the localities which were molten at the time of application of the augmented strain. Thus hot cracks, often seen in welds adjacent to the fusion line in 70 copper-30 nickel, could be readily nucleated in the partially melted region.

According to the hypothesis of Fig. 1, there are two ways of reducing the extent of the partially melted region:

1. Employ conditions which would produce a steeper thermal gradient, such as welding at faster travel speeds or adding chills to increase the steepness of the temperature gradient.
2. Weld on more homogeneous material. The solute concentration level is probably not as important as how evenly the solute is distributed. It would be desirable to eliminate low melting localities and have all regions of the material exhibit the same solidus-liquidus range. However, definite practical limitations prevent production of such a perfectly homogeneous material.

The Varestraint test should be an effective tool for evaluating the two suggestions above. In the first instance, relative hot cracking at the fusion line could be correlated to various welding conditions. In the second case, part of a heat of material could be subjected to extensive hot working in an attempt to break up the solute segregation present. The hot cracking tendencies of hot worked material could then be compared to the properties of the same material cold reduced an equivalent amount with a minimum number of intermediate annealing operations.

Conclusions

1. A partially melted region occurs adjacent to the fusion line of welds made in a commercially prepared alloy of 70 copper-30 nickel.

2. Welds made in 70 copper-30 nickel exhibit solute segregation in the partially melted region.

3. The superposition of a curve showing the distribution of peak temperatures adjacent to the fusion line upon the variations in the effective melting temperature, resulting from solute segregation inherent in commercially processed materials, provides the basis for a hypothesis explaining both the mechanism and extent of the partially melted zone.

4. Metallographic evidence of partial melting includes all of the features predicted by the hypothesis, namely: (a) localized melting to form "dots" within the matrix of grains, and (b) interaction between grain boundaries and localized molten regions to cause both grain boundary broadening and interference with normal grain growth.

5. Electron beam microprobe analysis of the broadened grain boundaries and "dots" in the partially melted region shows that these localities invariably exhibit solute segregation of a form which causes localized reduction of the effective melting temperature.

6. The partially melted region in welds of 70 copper-30 nickel is probably a nucleation site for hot cracking in the base metal adjacent to the weld fusion zone.

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