

# **Joining Metals Using Semi-Solid Slurries**

by

**Patricio F. Mendez**

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University of Buenos Aires, Argentina

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Signature of Author .....  
Department of Materials Science and Engineering  
January 20, 1995

Certified by .....  
Stuart B. Brown  
Richard P. Simmons Associate Professor of Materials Manufacturing  
Thesis Advisor

Accepted by .....  
Carl V. Thompson II  
Professor of Electronic Materials  
Chair, Departmental Committee on Graduate Students

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## **Abstract**

A method of joining metals with semi-solid slurries is presented. Experiments have been carried out using a model alloy of Sn-Pb to demonstrate the concept. The advantages of this process are a controlled microstructure of the weld, lower post-welding stresses and a small heat affected zone. Tensile tests have been performed, showing a weld strength comparable to bulk material strength. Key factors in this process are the substrate and slurry temperature as well as the absence of superficial oxides or contaminants. The substrate must be preheated locally before the slurry fills the weld groove so that the portion of the substrate in contact with the oncoming slurry is also in the semi-solid state. The process described here has a patent pending in the U.S. Office of Patents and Trademarks.

Thesis Supervisor: Stuart B. Brown

Title: Richard P. Simmons Associate Professor of Materials Manufacturing

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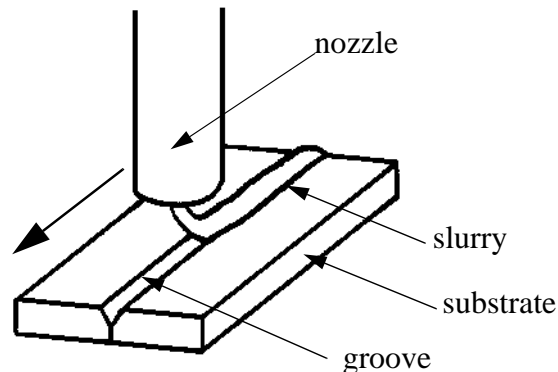
# Joining Metals Using Semi-Solid Slurries

Patricio Mendez

Department of Materials Science and Engineering  
Massachusetts Institute of Technology

## 1. Introduction

Joining metals with semi-solid slurries consists of depositing a continuous stream of fully dense semi-solid slurry along a preheated weld groove as shown in Figure 1.



**Figure 1:** Welding using semi-solid slurry

In contrast to welding using semi-solids, the most commonly used welding methods include a local heating of the substrate above the liquidus temperature (arc welding in all forms, laser and electron beam welding) [1, 2]. The liquid metal generally solidifies with a dendritic microstructure. This effect is not desirable from a metallurgical point of view because of the lack of control of the microstructure, which can possess porosity, inclusions and uncontrolled grain orientations. Also, the high temperatures necessary to completely melt parts of the substrate induce a heat affected zone. In this zone the material undergoes a thermal history that makes its properties different and generally worse than those of the rest of the bulk material in the substrate, often lowering fracture toughness, corrosion resistance and yield strength. The decrease of fracture tough-

ness facilitates the appearance of cracks along the weld due to thermal and residual stress.

During the solidification of metal alloys, one can observe either a dendritic solid phase or a suspension of spheroidal solid particles within a molten phase. The type of solid phase is a function of the processing conditions (cooling and shear rates). Alloys with this spheroidal microstructure in the semi-solid state are called semi-solid metal slurries, or just semi-solid slurries.

Welding with semi-solid slurries offers the potential of avoiding the problems mentioned above because the solidification and heat transfer processes are radically different than those of welding using a purely liquid phase. In welding with semi-solids the solidified microstructure is globular (not columnar dendritic as in the other types of welding mentioned above) because in a semi-solid slurry solidification starts at all solid particles simultaneously. The heat affected zone is greatly decreased in welding with semi-solid fillers because it is not necessary to completely melt the material near the joint, and the filling material is at a relatively low temperature. Semi-solid welding also dramatically decreases welding distortions. The temperature difference between the weld pool and the bulk substrate is much smaller than in common welding since the slurry temperature is below the liquidus temperature.

In welding using semi-solid slurries, the filler is deposited as a continuous stream, and not by droplet deposition as in arc welding. Since the semi-solid slurry is fully dense, so will be the weld bead, dramatically reducing the porosity that can form in other kinds of welding. Semi-solid slurries allow for the control of the apparent viscosity of the deposition material in such a way that the filler remains where it was applied and does not flow along the weld groove, even for large filling cross sections. This viscosity control is not possible with liquids. A comprehensive description of the fundamentals of semi-solid slurries behavior is given in references 3, 5 and 6, and a dimensional analysis of the deposition process is presented in Appendix I.

Since semi-solid welding does not involve electric currents for deposition (as in arc or resistance welding), the substrate can be a non-conductive material such as ceramics. The present limitation is in the production of a high temperature semi-solid slurry, not in the concept of joining materials with semi-solid slurries.

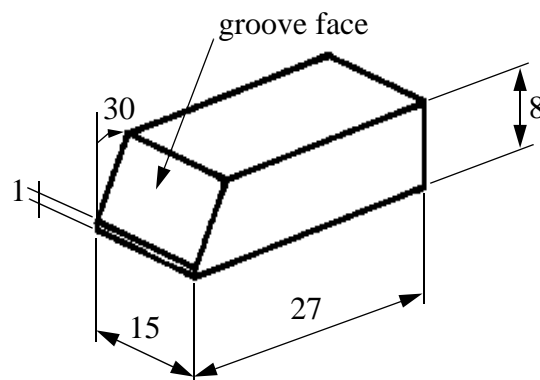


With semi-solid welding there is no high emission of radiation in the UV wavelength since no arc is involved. This process is of a continuous nature capable of welds of virtually any length.

An air atmosphere was used in the experiments performed, and satisfactory welds were obtained. However, for other materials with greater affinity with oxygen, an inert atmosphere or the presence of a slag might be necessary. Using an inert atmosphere instead of a slag would be beneficial, as in that case no post-weld cleaning would be necessary.

## 2. Description of the Experiments

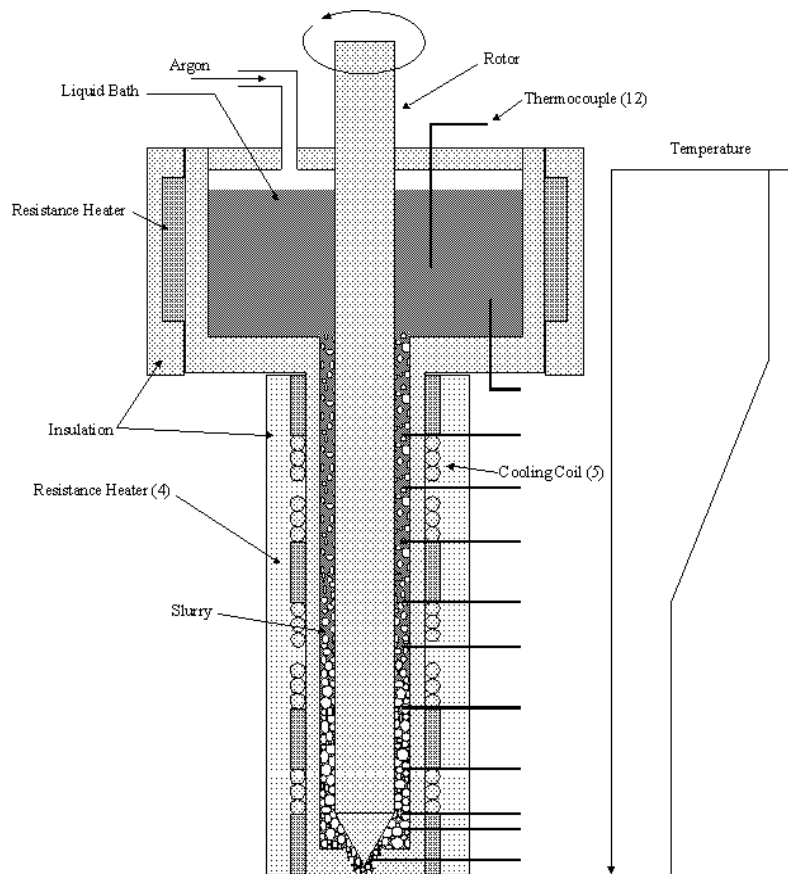
In our experiments we welded bars of Sn 85%-Pb 15% (Figure 2) with a semi-solid slurry composition of approximately Sn 95%-Pb 5% measured both by density and by determining the liquidus temperature. The bars to be welded were mounted on a computer-controlled x-y-z table with a closed loop controlled heating plate. The substrate temperature and the deposition velocity could be controlled accurately this way.



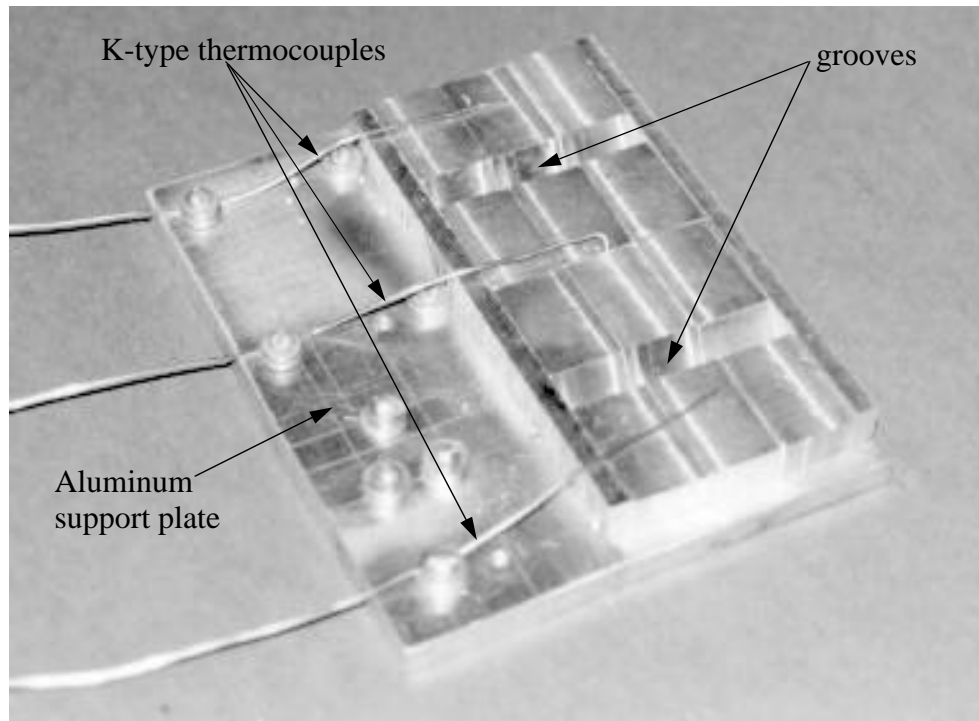
**Figure 2:** Dimensions of bars welded (in mm)

The semi-solid slurry was produced in a rheocaster developed by Chris Rice and Stuart Brown shown schematically in Figure 3, (from Rice's work [4]). The bars were fitted with K-type thermocouples to record temperature histories. The bars are mounted on an aluminum plate, Figure 4, and the whole arrangement is mounted on a heating plate. The bars had an as-cast dendritic microstructure that coarsened slightly during the heating stage (approximately 35 minutes to reach

the target temperature). The slurry had a solid fraction of 51% and a typical solid particle diameter of 100 microns, giving the slurry an apparent viscosity, or deformation resistance, of the order of 2.4 Pascal-seconds [5]. The displacement velocity used was 20 mm/s, and the slurry flow rate was  $1.6 \text{ cm}^3/\text{s}$ . The best results were obtained with a substrate temperature of  $198^\circ \text{C}$  and a slurry temperature of  $218^\circ \text{C}$ . The experiments welded two sets of three pairs of bars for each run of the rheocaster. Figure 4 shows six pairs of bars arranged for welding. The weld was performed by depositing a slurry in a rectilinear path along the weld grooves. After welding the first set of three pairs of bars, the slurry temperature and liquid fraction from the rheocaster were allowed to increase slightly before welding the second set.



**Figure 3:** Rheocaster developed by C. Rice and S. Brown (drawing taken from ref. 4)



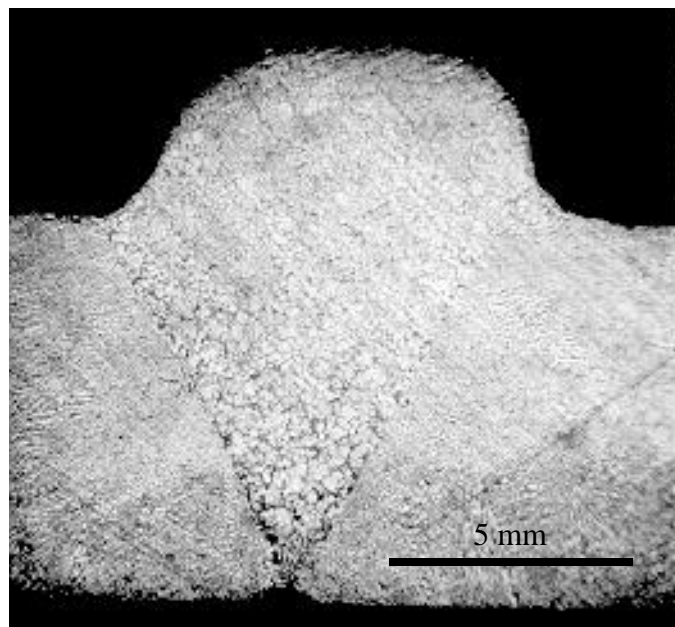
**Figure 4:** Set-up of the bars to be welded

The model material employed, Sn 85%-Pb 15%, was selected as the low temperature system that has been typically used for semi-solid experiments. [4-6]. The rheocaster was also loaded with an alloy of this composition. Due to density differences between the liquid phase (Pb rich), and the solid phase (Sn rich), separation of both phases happened to a certain extent. The consequence of this is that at the beginning of the slurry production, the flow obtained was a liquid Pb rich phase, followed by a more or less steady semi-solid slurry of a lower Pb fraction. Density and liquidus temperature measurements estimate this slurry composition at Sn 95%-Pb 5%.

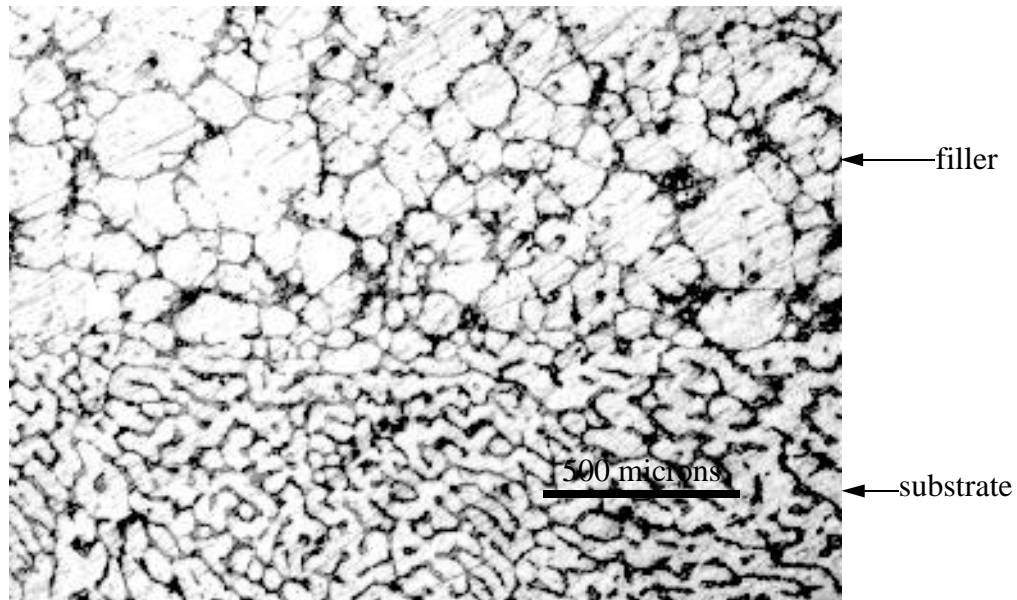
### 3. Results

Figure 5 is a micrograph of two welded bars, conveniently etched to show the difference in microstructure between the bars and the filler. The bars have a coarsened dendritic microstructure. The semi-solid filling, in contrast, has a globular microstructure where the globules are the solid particles that grew and coarsened during cooling. Between the globules there is Sn-Pb eutectic. Figure 6 shows a close-up of the joint between the substrate (bar) and the filler (semi-solid slurry).

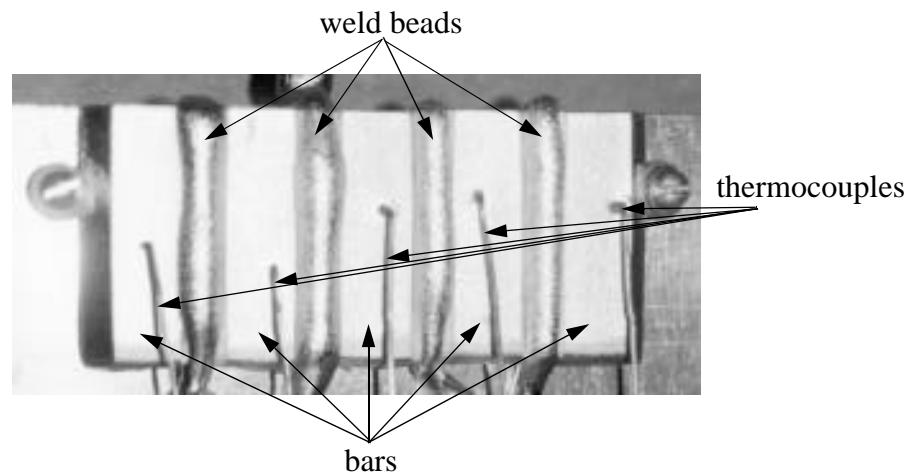
The substrate is in the bottom part of the picture and the semi-solid filler on top. It can be seen that both microstructures are significantly different, and there is a fast transition from one to the other, yet without the presence of an interface or an oxide layer. The microstructure gradually changes its morphology in a space of 300 microns. It is remarkable that we do not see either a heat affected zone nor micro cracks or porosity. The appearance of the weld is also very smooth and not wrinkled, as can be seen in Figure 7. This figure shows an early experiment setup, with a different arrangement of the bars than in Figure 4.



**Figure 5:** Micrograph of two bars welded with semi-solid slurry. Magnification: 6.25x.



**Figure 6:** Joint between the semi-solid slurry and the dendritic substrate. Magnification: 50x.



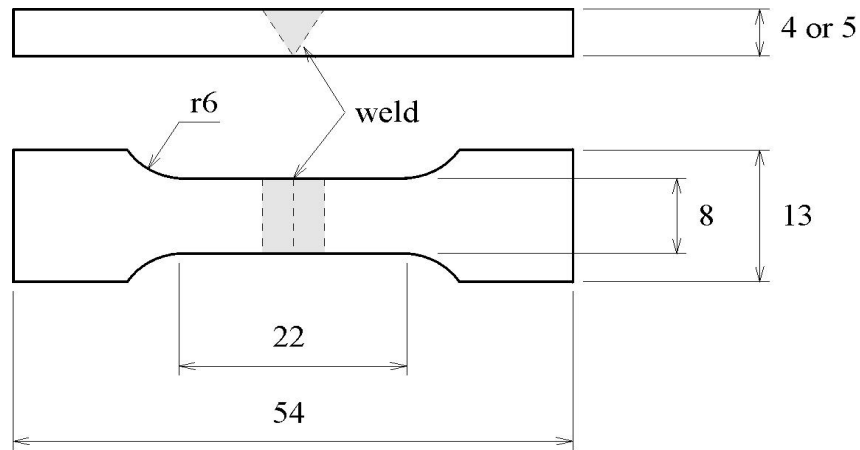
**Figure 7:** Weld beads showing a smooth surface.

Tensile tests have been performed on specimens welded with this process. A displacement controlled tensile machine was used. The tensile specimen geometry is shown in Figure 8. The displacement rate was 1 in/min, approximately equivalent to a strain rate of  $1.9 \times 10^{-2}$  in/in/s. This

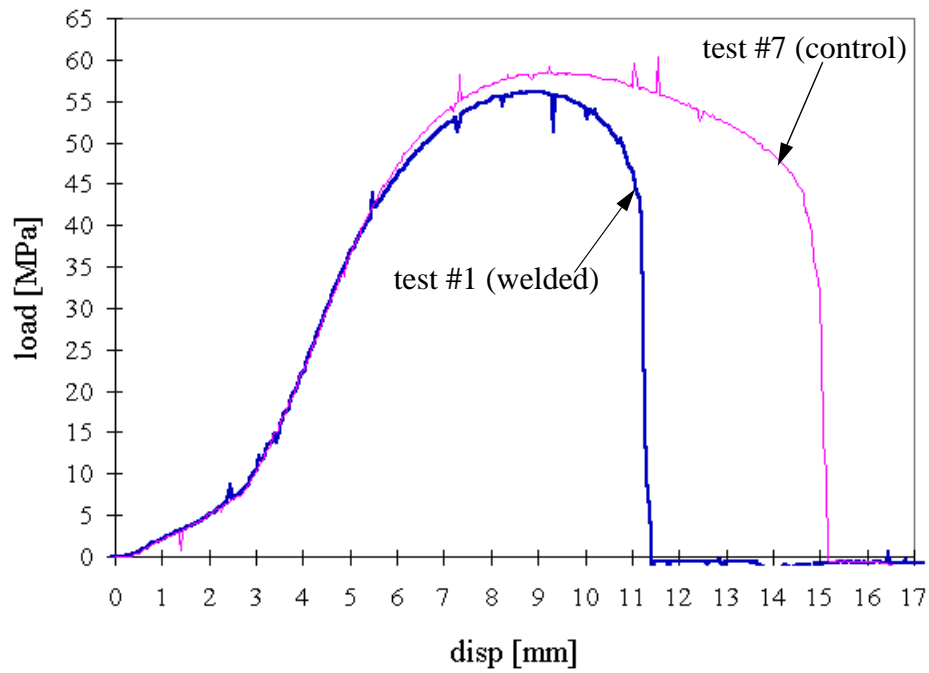
high strain rate was chosen to reduce rate-dependent effects, since room temperature corresponds to a high homologous temperature for the alloy tested. The results obtained are presented in Table 1. Figure 9 shows superimposed load/displacement curves for tests #1 (welded) and #7 (control), with 4 mm thickness tensile specimens.

**Table 1: Results of the Tensile Tests**

Test #	UTS	Thickness	Comments
	[MPa]	[mm]	
1	56.0	4	
2	55.4	4	
3	54.9	4	
4	55.3	5	
5	52.9	5	
6	48.4	5	
7	58.5	4	control specimen, not welded
8	59.5	5	control specimen, not welded
9	49.2	5	control specimen, not welded
<b>Average not welded</b>	<b>55.7 max +3.8 min -6.5</b>		
<b>Average welded</b>	<b>53.8 max +2.2 min -5.4</b>		



**Figure 8:** Dimensions of specimens used for tensile tests (in mm)



**Figure 9:** Load-displacement curves for welded and control specimens.

Some specimens exhibited pitting in the surface of the gauge length after machining. We discarded these specimens because of the sensitivity of the tensile test result to stress concentra-

tions. It was observed that cracks started in these surface pits at early stages of the tension test. One possible origin of this pitting is the as-cast microstructure of the bars welded. Another possibility is that the pitting originated from surface contaminants or oxidation on the face of the weld groove before the deposition of the slurry. Despite that in Figure 6 there is no contaminant or oxide film, there is a possibility that some parts of the exposed surface were contaminated. This problem happened in about a third of the specimens. This effect would be reduced if the heating of the substrate is carried out in an inert atmosphere.

#### **4. Discussion**

It was seen during the experiments that this process is very sensitive to the temperatures of the slurry and substrate and the corresponding liquid fractions. The exact mechanism of the metallurgical joint between the slurry and the substrate is not yet well understood. For the temperature and composition of the substrate (198 °C, Pb 15%), the corresponding liquid fraction is 57% [6]. For the temperature and composition of the slurry (218 °C, Pb 5%), the liquid fraction is 49%. Although the substrate has a higher liquid fraction than the slurry, it still keeps its shape during the time of the experiment while the slurry deforms significantly under its own weight. The slurry can flow more easily because it has a globular structure whereas the substrate has higher deformation resistance because it has a dendritic structure that has to be disassembled for large deformations to occur [9].

A heat balance for the interface during the first moments of contact between the slurry and the substrate gives an interface temperature of 210 °C (Appendix III). During these first moments, the substrate side of the interface is fully liquid or has a very low solid fraction (the liquidus temperature for the composition of the substrate is 208 °C [6]). This “soft-interface” mechanism would permit the solid particles of the slurry settle gradually over the dendritic substrate without aligning them along the surface of the substrate, what would mark an interface. This mechanism could be the reason of the excellent metallurgical joint observed in our experiments. Additional work on this welding process will be helpful to validate this hypothesis.

#### **5. Future efforts**

With the experiments performed we proved the feasibility of using semi-solid slurries for



joining metals. Future efforts include the application of this technology to alloys of commercial interest with higher liquidus temperatures and better structural qualities, such as ferrous alloys or aluminum alloys. Further work is also required to determine the best process for preheating the weld groove and to prevent groove face oxidation or contamination. One possible method is to pre-heat the weld groove with a hot inert gas or plasma immediately before depositing the slurry. With this preheating method there is also a protective atmosphere and absence of slag. Practical considerations also remain to be addressed, especially from the point of view of design for ease of use.

## **6. Conclusions**

- It is possible to weld metals with semisolid slurries. The advantages of this process are a controlled microstructure of the weld, lower post-welding stresses and a small heat affected zone.
- The critical parameters for obtaining a good weld are the substrate and slurry temperature as well as the absence of superficial oxides or contaminants.
- Semi-solid fillers provide the unique feature of having a controlled flow once deposited. Also operating temperatures and thermal gradients are smaller than in arc welding.

Semi-solid welding is a significant innovation in welding. We believe that the process presented here will overcome some problems of existing welding methods and will allow new welding applications not existing today.

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## Appendix I: Dimensional Analysis

The Peclet number ( $Pe$ ) gives an indication of the ratio between the heat transferred due to motion of the heat source and heat transferred by conduction in the direction of motion. Its expression is:

$$Pe = \frac{VL}{\alpha} \quad (I-1)$$

where:

$V$  = relative velocity of nozzle (0.02 m/s)

$L$  = characteristic length in the direction of welding ( three bars of 15 mm width: 0.045 m)

$\alpha$  = thermal diffusivity of material

Since the semi-solid slurry is a two-phase flow, in which the liquid phase is solidifying, the thermal diffusivity of this material needs some special consideration. The thermal conductivity of the slurry can be estimated approximately as 50 W/m/K [7]. The density of the alloy can be calculated using:

$$\frac{1}{\rho} = \frac{\text{wt}\%_{Sn}}{100\rho_{Sn}} + \frac{\text{wt}\%_{Pb}}{100\rho_{Pb}} \quad (I-2)$$

From equation I-2 we obtain  $\rho = 7438 \text{ kg/m}^3$ . For the heat capacity:

$$dH = f_s c_{ps} dT + f_l c_{pl} dT + \Delta H_{ls} df_s \quad (I-3)$$

Also:

$$f_s = \frac{x_l - x}{x_l - x_s} \quad (I-4)$$

$$x_l = \frac{T - T_m}{m} \quad (I-5)$$

$$x_s = kx_l \quad (I-6)$$

Combining these equations, we obtain:

$$c_p = \frac{dH}{dT} = f_s c_{ps} + f_l c_{pl} + \frac{\Delta H_{ls}}{T_m - T} \left( \frac{1}{(1-k)} - f_s \right) \quad (\text{I-7})$$

In our experiments:

$f_s \approx 0.51$  : solid fraction

$f_l \approx 0.49$  : liquid fraction

$c_{ps} = 228$  J/kg/K: heat capacity of solid

$c_{pl} \approx 66$  J/kg/K : heat capacity of liquid (estimated from experiments)

$\Delta H_{ls} \approx 58.52$  kJ/kg : enthalpy of solidification [5]

$T = 218$  °C: slurry temperature

$T_m = 232$  °C: melting temperature

$k \approx 0.058$  : partition coefficient (estimated at eutectic temperature)

$m \approx 1.3$  °C/wt% Pb : liquidus slope

Using these values we obtain  $c_p \approx 2454$  J/kg/K,  $\alpha \approx 2.74 \times 10^{-6}$ , and  $Pe \approx 329$ . This value of  $Pe$  is much larger than 1, indicating that the conduction of heat in the longitudinal direction is negligible in a coordinate system fixed to the nozzle. The heat flow from the slurry to the bulk material can then be considered approximately normal to the weld interface.

The Biot number ( $Bi$ ) gives an indication of the effect of convective cooling on temperature homogeneity in the weld bead. Its expression is:

$$Bi = \frac{hl}{k} \quad (\text{I-8})$$

where:

$h \approx 20 \text{ W/m}^2 \text{ K}$  : convection coefficient for still air, considering radiation

$l = 0.012 \text{ m}$ : distance from bottom to top of the weld bead

$k \approx 50 \text{ W/mK}$  : thermal conductivity, estimated from ref. 7

Substituting numerical values we obtain  $Bi = 0.0048$ . This small value of  $Bi$  indicates that there is a negligible effect of convective cooling in the temperature distribution of the weld bead. Any temperature gradient within the weld bead will be due to heat transfer between the slurry and the interface. Once the temperature is homogenized, all points of the filler will cool down at the same rate.

We propose a dimensionless group that characterizes the deposition of a viscous material. This parameter evaluates the importance of deformation due to own weight relative to the deformation resistance given by the viscosity. For equal values of this parameter the time dependent deformation due to weight of any two materials is similar. The dimensionless group is expressed as:

$$Me = \frac{\rho g t L}{\mu} \quad (\text{I-9})$$

where:

$\rho$  = density

$g$  = acceleration of gravity

$t$  = characteristic time

$L$  = characteristic dimension

$\mu$  = apparent viscosity

Values of  $Me$  much larger than one indicate liquid-like behavior, much smaller than one, solid-like behavior, and not far from one is the typical range for semi-solids and pastes. The larger

the magnitude of this parameter, the more fluid is the slurry. We can now estimate typical values of  $Me$  in our welds. For the semi-solid slurry deposited:  $\rho = 7438\text{kg/m}^3$ ,  $g = 9.81\text{m/s}^2$ ,  $L = 0.012\text{m}$ . From temperature records a characteristic time can be estimated as  $t \approx 5$  seconds. The following empirical formula fits very well the data determined by Kumar for a globular semi-solid structure between  $f_s = 0.30$  to  $0.45$  ( ref. 5, page 111):

$$\mu = 8.5787 \times 10^{-2} f_l^{-4.642} \quad (\text{I-10})$$

where:

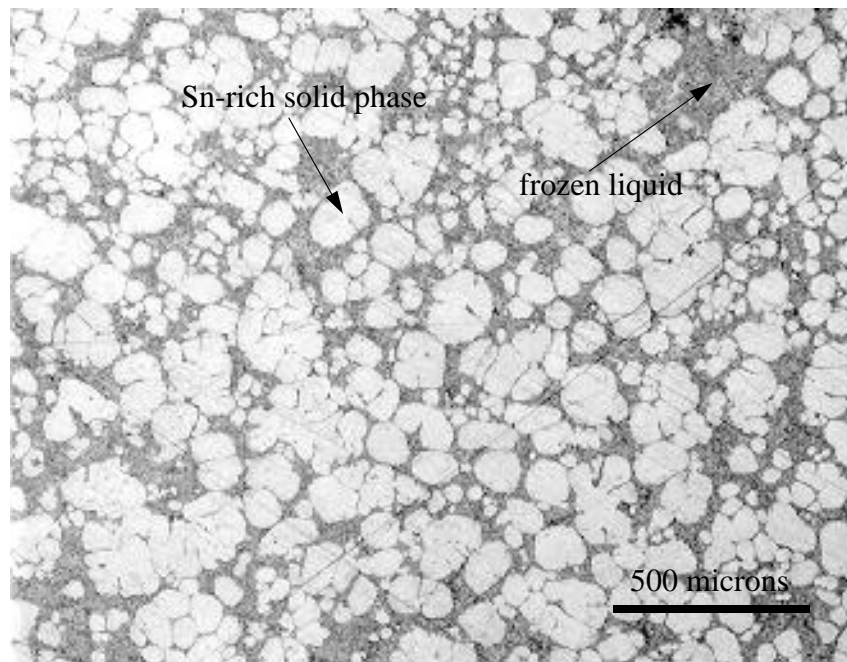
$\mu$  = apparent viscosity in Pascal-seconds

$f_l$  = liquid fraction in kg/kg

For a slurry of  $f_l=49\%$  we obtain  $\mu=2.4$  Pascal-seconds. The value obtained is  $Me \approx 365$ . This high value indicates fast deformation of the slurry but limited flow, in agreement with what was seen in our experiments. If instead of a slurry we used molten metal, its viscosity would be  $\mu \approx 10^{-3}$  Pascal-seconds [3], then  $Me \approx 9 \times 10^5$ . In this case the flow would be controlled by forces other than viscous, e.g. surface tension.

## Appendix II: Microstructural Evolution of the Weld

One interesting result of our experiments indicated that particles that constituted the solid fraction grew with a stable front, contrary to what is commonly expected. We carried out experiments freezing the microstructure coming out of the nozzle by quenching the slurry in ice (Figure II-1). In this figure what appears white is the Sn-rich phase that constituted the solid fraction, and what is gray is the frozen liquid phase. In Figure 6, where the slurry cooled down more slowly, the composition of the phases is close to equilibrium, where the white zones are Sn-rich phase and the gray zones are Sn-Pb eutectic.



**Figure II-1:** Frozen semi-solid slurry quenched on ice. Magnification: 50x.

Comparing Figures 6 and II-1, one sees a higher proportion of the Sn-rich phase in Figure 6 (slowly cooled-down weld) than in Figure II-1 (frozen semi-solid microstructure) implying a growth of the Sn-rich phase. Micrographs with higher magnification show that the particles of Figure 6 grew with a stable front. We believe that this stable growth is due to surface energy effects and the slow cooling rates experienced during our welding experiments. If we add the contribution of the surface energy by using a simplified Mullins-Sekerka criterion for a planar solid-liquid in-

terface [8], the resulting equation indicates that stable front growth is possible for small particles when:

$$\frac{dT}{dx} > \frac{mVC_L^*(1-k)}{D} - \Gamma\left(\frac{2\pi}{\lambda}\right)^2 \quad (\text{II-1})$$

where:

$dT/dx \approx 0$ : temperature gradient normal to the solid/liquid interface.

$m \approx 1.3 \text{ }^\circ\text{C/wt\% Pb}$ : liquidus slope

$V \approx 4 \times 10^{-6} \text{ m/s}$ : growth velocity

$C_L^* \approx 38.1 \text{ wt\% Pb}$ : liquid composition at the interface (eutectic)

$k \approx 0.058$ : partition coefficient (eutectic)

$D \approx 6.2 \times 10^{-9} \text{ m}^2/\text{s}$ : diffusivity of Pb in Sn [5]

$\Gamma \approx 7.85 \times 10^{-8} \text{ K m}$ : Gibbs-Thompson coefficient [5]

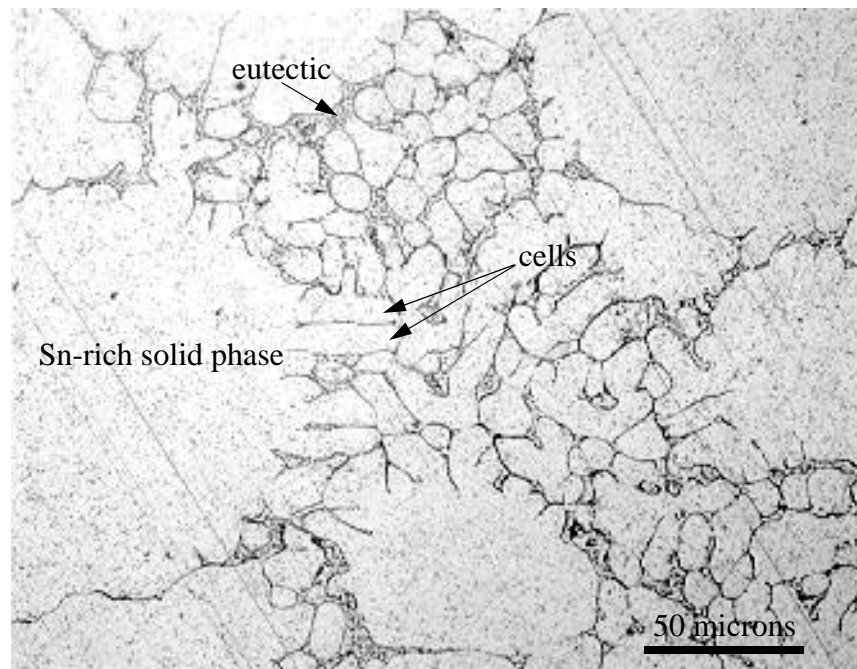
$\lambda$ : wavelength of a surface undulation

The thermal gradient can be neglected because of the small temperature difference between the substrate and the slurry that allows a fast temperature homogenization. The growth velocity is estimated as the distance travelled by the growth front in the characteristic time. From Figure II-1, a typical gap between two solid particles in the liquid is 40 microns and from temperature records we can estimate the characteristic time in 5 seconds. This gives a growth velocity of 4 microns/s for each of the opposite growing fronts.

We see in equation 10 that for small particles the growth is stable, since the allowed wavelengths—those shorter than the perimeter—decrease with size of the particle. Slow cooling rates also favor a stable front growth. Solving equation 10 for  $\lambda$ , we obtain a wavelength of 10 microns, smaller but very close to perimeter length (for this order of magnitude approximation), suggesting



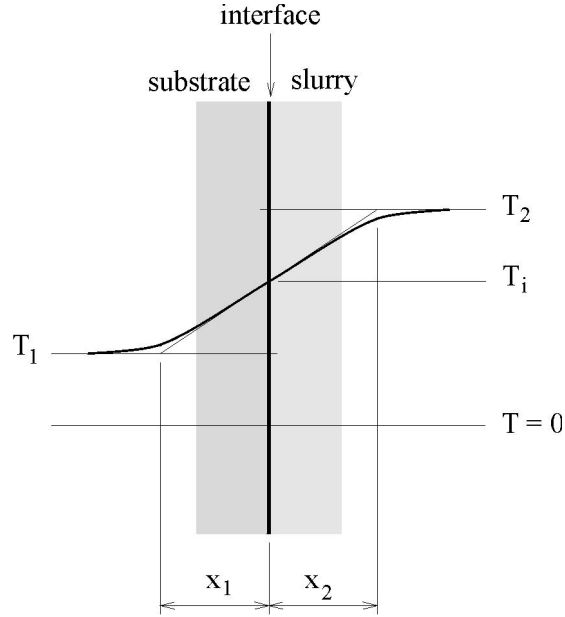
that at the current rate of cooling there is a strong possibility of stable front growth. In Figure II-2, the rate of cooling was increased by pouring the slurry on a metallic plate instead of a hot weld groove, and the beginning of cellular growth is observed.



**Figure II-2:** Beginnings of cellular growth on a slurry cooled on a thin metallic plate.  
Magnification: 400x.

### Appendix III. Slurry-Substrate Interface Initial Temperature

A schematic of the temperature profile during the initial moments of contact between the slurry and the substrate is shown in Figure III-1. For short times and similar thermal conductivities this temperature profile can be approximated as a straight line as shown.



**Figure III-1:** Temperature at the interface during the first instants of contact of the slurry with the substrate

An energy balance in the vicinity of the interface indicates that the heat lost by the slurry is absorbed by the substrate. This can be expressed as:

$$\frac{1}{2} (T_2 - T_i) x_2 \rho_2 c_{p2} = -\frac{1}{2} (T_1 - T_i) x_1 \rho_1 c_{p1} \quad (\text{III-1})$$

where we used subindexes 1 and 2 to denote properties of the substrate and slurry respectively,  $T_i$  is the interface temperature. The continuity of the slope of the temperature profile at the interface indicates:

$$\frac{T_2 - T_i}{x_2} = -\frac{T_1 - T_i}{x_1} \quad (\text{III-2})$$

Equations (III-1) and (III-3) combined give the final equation that relates the interface temperature to the slurry and substrate temperature. The specific heats can be calculated with equation I-7 of Appendix I.

$$\rho_2 c_{p2} (T_2 - T_i)^2 = \rho_1 c_{p1} (T_1 - T_i)^2 \quad (\text{III-3})$$