The High-Temperature Wetting Balance and the Influence of Grit Blasting on Brazing of IN718

Different grit blasting materials and mechanical polishing were compared to determine their influence on wetting

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ABSTRACT. A high-temperature wetting balance was developed to provide information about the wetting force and its time dependence. The instrument and the development of the wetting force are discussed. As example of the operation of this instrument, data are presented on the effects of grit blasting of IN718 on the wetting by AMS4777 (AWS BNi-2). The grit blasting was done with Al_2O_3, SiC, and a Ni-Cr-Fe alloy, and contrasted with mechanical polishing. Grit blasting with Al_2O_3 hinders subsequent wetting, but mechanical polishing of this grit-blasted surface restores the wettability. Grit blasting with the Ni-Cr-Fe alloy did not impede subsequent wetting. The effect of grit blasting with SiC was intermediate compared to that of the other two media. The measured time dependence of wetting stems from the time dependence of the chemical reactions (such as the dissolution of oxides, the diffusion of braze constituents into the substrate, and the development of intermetallic compounds) that occur when a substrate reacts with a liquid braze.

Introduction

Wetting balances have been used for many years to characterize the wetting behavior required for high-quality solder and brazed joints (Refs. 1–11). Most of these studies (Refs. 1–8) have been restricted to soldering because the commercial instruments do not operate at the elevated temperatures required for brazing, and they do not have the capability to operate in the inert environments that are generally required. This paper describes, in some detail, a simple wetting balance built for brazing studies. The classical interpretation of the wetting force that is

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measured by the balance is discussed, as are newer approaches that account for the chemical reactions that generally accompany the wetting of a metal by a liquid braze. These newer approaches explain the origin of the time-dependent behavior that is measured by the wetting balance.

The problem of surface preparation via grit blasting is addressed with the wetting balance. Different grit blasting media and mechanical polishing are compared in order to determine their influence on subsequent wetting.

Background

The wetting force concept was developed more than 100 years ago, and it adequately describes the physical wetting where chemical reactions do not play a role in wetting. More modern approaches to wetting take chemical reactions into account and the influence of these on the kinetics of wetting. The consideration of chemical reactions provides the means for explaining the origin of the time dependence of wetting that is measured by the wetting balance. This is an important consideration because one of the chief benefits to performing a wetting balance test is that it provides information on the wetting time, which is not obtained by the simple measurement of the wetting angle of a drop solidified onto a substrate. The wetting balance also provides quantitative

KEY WORDS

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data on the magnitude of the wetting force.

The classical interpretation of the wetting force is that of a force developed by the balance of surface tensions acting on a liquid in contact with a solid surface. It assumes no chemical interaction of the liquid and the surface. The surface tension is defined as the change in free energy of a system as a function of the change in area of a boundary between phases. It has units of an energy/area or force/length. The equilibrium of the various forces/length that arise when phases are in contact can be treated by the simple mechanical balance of forces shown in Fig. 1.

The wetting angle, θ, is defined by the balance of the forces/lengths developed between the solid, liquid, and the surrounding vapor where γ_{SL} is the interfacial tension between the liquid and the solid, and γ_{SV} and γ_{LV} are the surface tensions between the solid and the vapor and the liquid and the vapor, respectively. This balance of surface forces yields

$$\gamma_{SV} = \gamma_{SL} + (\gamma_{LV} \cos \theta) \qquad (1)$$

The difference between γ_{SV} and γ_{SL} has been termed the wetting force, which represents the difference in the energy/area (or the force per unit length) when a given area of solid, in contact with a vapor, is replaced by a liquid. Correcting for the perimeter, L, to give a force F, rather than a force per unit length, gives

$$F = L (\gamma_{SV} - \gamma_{SL}) = L \gamma_{LV} \cos \theta \qquad (2)$$

The magnitude of the wetting force is important because this force controls capillary action (for instance, the force which draws the liquid braze into a gap between parallel plates) and is, along with the viscosity, the physical property that determines the time required for wetting (Ref. 3). Equation 1 was developed from purely mechanical arguments by Young (Ref. 12)

and Dupré (Ref. 13), and is, therefore, referred to as the Young or Young-Dupré equation. This expression can also be derived from thermodynamics (Refs. 10, 14–29).

These mechanical and the earliest thermodynamic models considered the solid-liquid interface as having zero thickness with no compositional mixing within the layer or chemical reaction between the solid and liquid. This is wetting in the absence of surface chemical reactions. Reactive wetting, which considers surface reactions, has been described by more modern approaches (Refs. 10, 22–29). These reactions include the development of boundary layers in the braze and solid being brazed, diffusion into these layers, oxidation and reduction reactions at the surface, and, most importantly, the formation of intermetallic phases at the solid-liquid interface. Brazing almost always involves this reactive wetting. Reactive wetting is a dynamic process, as the reaction times govern the development of the wetting force and the time required for brazing. The wetting balance measures this wetting time, as well as the magnitude of the wetting force. Generally (Ref. 10), the energy/area of the surface reaction is much larger than the conventional definition of the solid/liquid interfacial energy. For instance, Yost and Romig (Ref. 26) have shown that for Pb/Sn solder wetting Cu, the energy/area associated with intermetallic phase formation is more than an order of magnitude greater than the interfacial surface energy in the absence of intermetallic phase formation. While the specific details of reactive wetting are beyond the scope of this paper, it is important to remember that the time dependence of the wetting force that is being measured is primarily due to the effects of surface reactions and their influence on γ_{SL}, and as Equation 2 shows on the measured wetting force. (The viscosity of the liquid can also introduce time dependence, but is not a problem for brazes because of their low viscosities.)

The Wetting Balance

A wetting balance (known in the physical chemistry literature as a Wilhelmy balance) is a device that measures the wetting force, F, being generated by a liquid wetting a solid. The device consists of a specimen (substrate), which is to be wet, suspended from a balance or load cell. A crucible containing the braze liquid is raised, eventually submerging the substrate by a predetermined amount. This creates a buoyancy force that pushes the sample upward, lessening the weight being measured. If wetting does not develop, the wetting force will also be negative and it
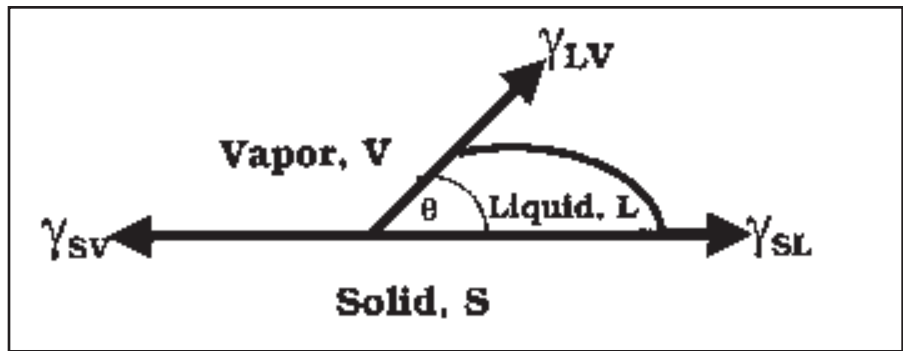

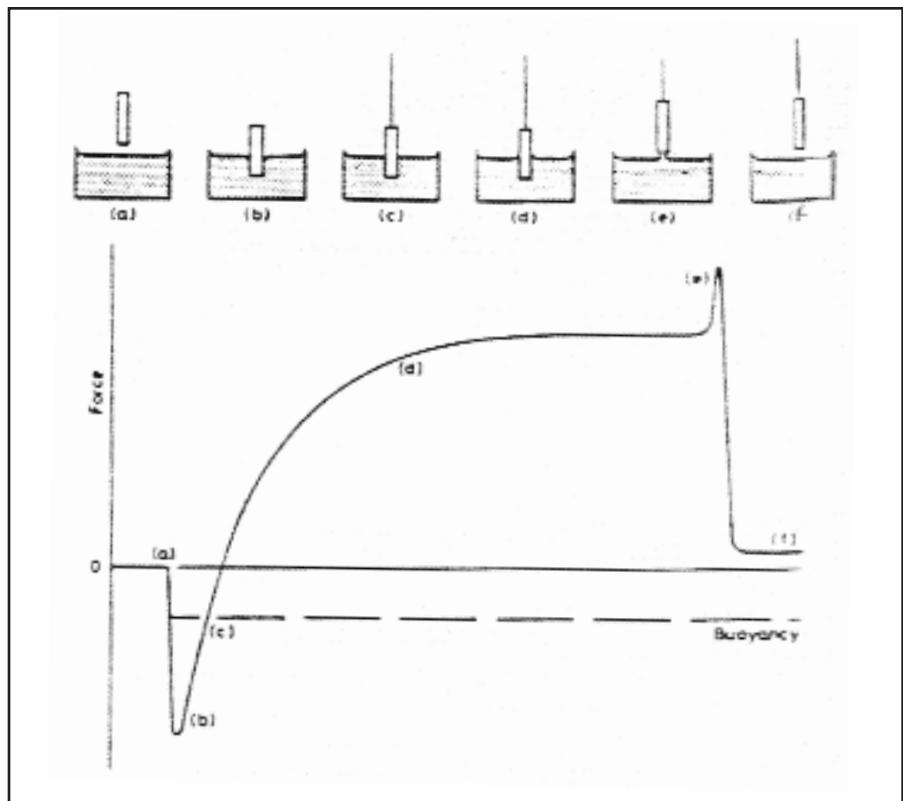*Fig. 1 — Balance of surface forces.*

Fig. 2 — Development of the wetting force (Ref. 2).

will act along with the buoyancy force to expel the sample from the liquid. (By convention, upward forces are considered negative and downward forces positive.) When, and if, a positive wetting force develops, the wetting force will pull the sample into the crucible, increasing the measured weight. The net force, F_N, measured by the balance is given by

$$F_N = L \gamma_{LV} \cos\theta - \rho_b g V_d \qquad (3)$$

where ρ_b is the density of the braze, g is the gravitational constant, and V_d is the volume of displaced liquid. $L \gamma_{LV} \cos\theta$ is positive when θ is less than 90 deg. F_N is positive when $L \gamma_{LV} \cos\theta > \rho_b g V_d$. From Equation 2, the wetting force per unit length of perimeter, F/L, is thus given by

$$F/L = \gamma_{LV} \cos\theta = (F_N + \rho_b g V_d)/L \quad (4)$$

Thus, the wetting force per unit length can be determined from F_N, the output from the weighing instrument. Equation 4 shows that a correction must be made for the buoyancy effect of the liquid that is displaced. The density of the liquid braze can be determined from literature values, or additional experiments. As shall be discussed, as the volume V_d, defined by the substrate area and the depth of immer-
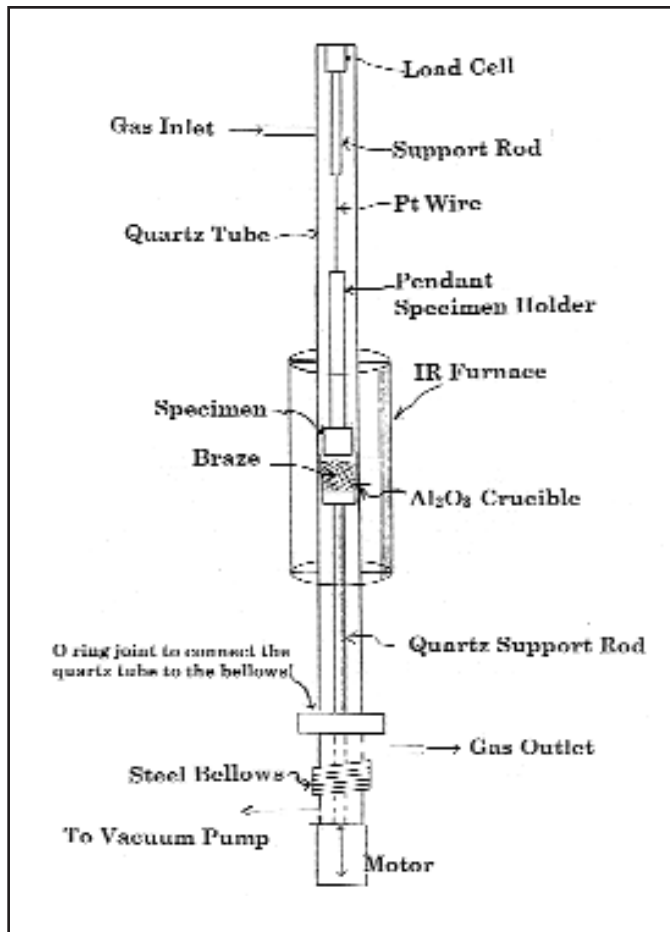
Fig. 3 — Schematic of the braze balance.

Fig. 4 — Wetting behavior of AMS4777 on IN718, which was grit blasted with Al_2O_3 prior to wetting. A — At 1075°C in 4.3 ⟨ 10^{-6} torr; B — at 1068°C in 5 ⟨ 10^{-6} torr.

sion, is generally small, the buoyancy correction is likewise small, so accurate determinations of the braze density are not crucial for the determination of the wetting force. If the surface tension, γ_{LV}, is measured separately, then Equation 2 can be used to determine the wetting angle, θ.

When a sample is immersed in a liquid, a meniscus will develop at the line of contact between the liquid and solid. The force defined by Equation 2 is the force developed by this meniscus. It has been shown (Ref. 30) that the force of the meniscus described by Equation 2 is valid even if the meniscus is curved, so long as the solid is perpendicular to the liquid and the wetting angle is constant around the perimeter. The perimeter L is that defined by the projection of the actual perimeter onto the plane of the liquid. Thus, if the meniscus is advanced or retarded in some places, this does not influence the measured value of F_N, or the calculated wetting force.

Figure 2 shows (Ref. 2) a typical wetting balance force record, with a downward force being defined as positive. This figure also shows the position of the crucible relative to the substrate, correlated with the measured force. The test starts with the crucible below the substrate (a). Immersion is accomplished by moving the crucible up at (b) and wetting occurs during (c) and (d). At (e), the crucible is lowered, with the substrate being completely out at (f). This typical wetting curve shows how F_N increases with time (b-e), reaching a steady state value at (d). (Figure 2 shows the net force, F_N. F/L is determined from the balance output shown in Fig. 2, and the corrections for buoyancy and L, as given in Equation 4.) The F_N curve of Fig. 2 defines the point in time (point c) where the measured force just equals that due to buoyancy, i.e., where wetting angle is 90 deg and the wetting for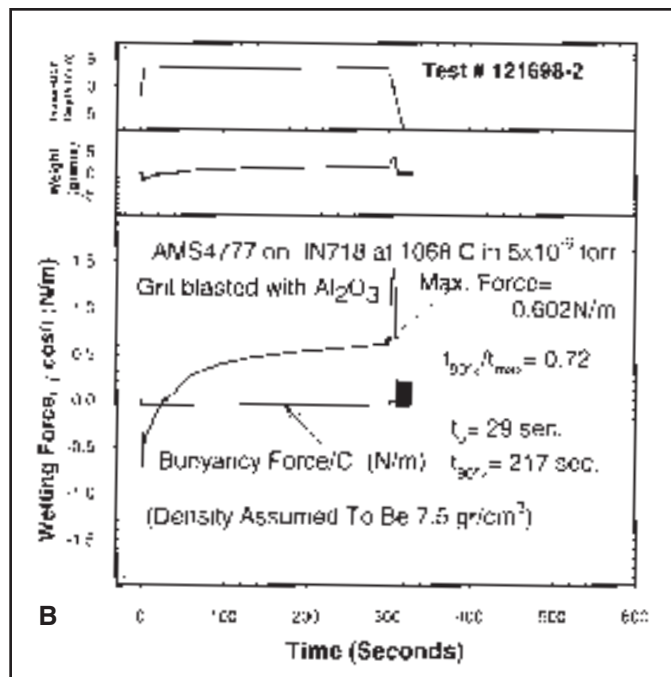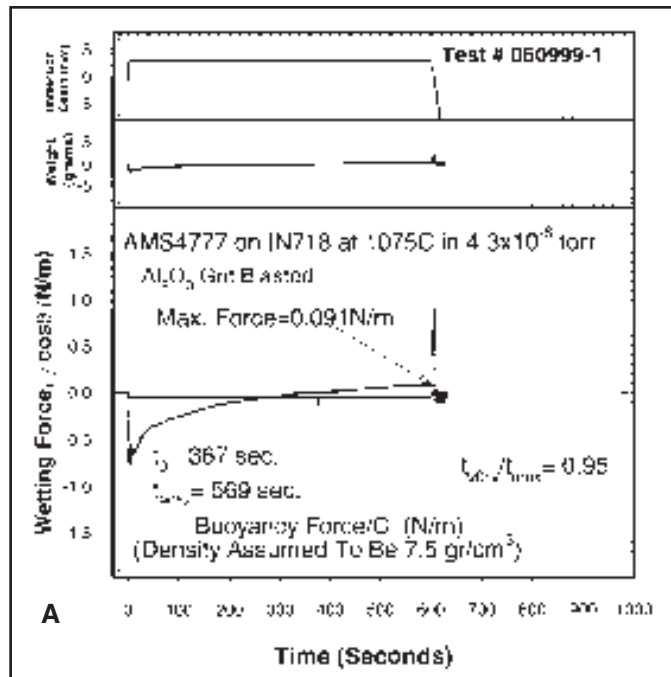ce, F/L, is zero. (The measured net force is still negative here because of the effect of buoyancy.) Increases in the net force shown in this figure are due to decreases in the wetting angle, or changes in γ_{LV}, if it is not constant. If γ_{LV} is assumed to be constant, then when the measured force increases above point c, it is because the wetting angle has decreased to below 90 deg.

Wetting balance measurements have been used primarily to describe liquid solder wetting a metal (Refs. 1–8). Here, this
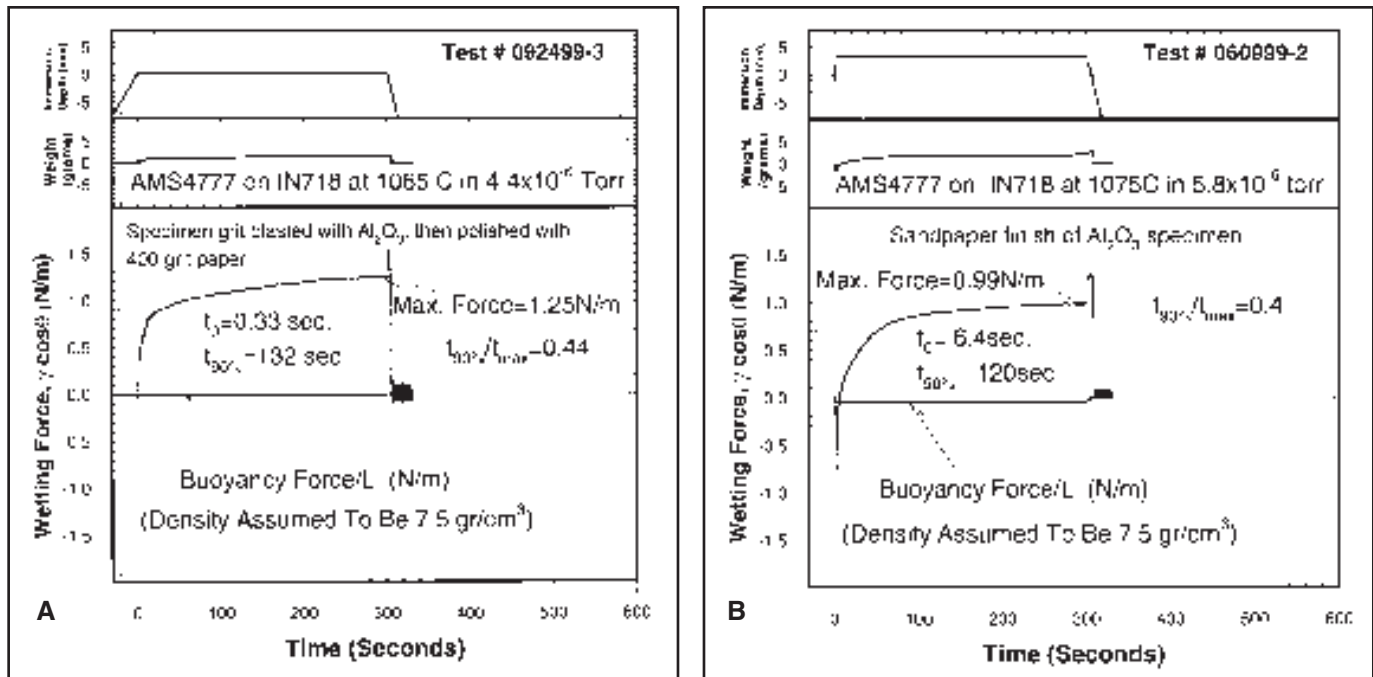
Fig. 5 — Wetting behavior of AMS4777 on IN718, which was polished with 400-grit paper after an Al_2O_3 grit blast. A — At 1065°C in 4.4 $\langle$ 10^{-6} torr; B — at 1075°C in 5.8 $\langle$ 10^{-6} torr.

approach is extended to the higher temperatures required for brazing.

The High-Temperature Wetting Balance

There are several commercial wetting balances available for the measurement of the wetting of solders, with a maximum temperature of operation of about 400°C. Furthermore, the solder and substrate are heated in air. For brazing, the temperature of operation must often exceed 1000°C and the substrate and braze must be heated in a controlled environment. A special wetting balance (Ref. 31), built to achieve these conditions, is shown in Fig. 3.

A Ti support rod is connected to the load cell and a fine Pt wire is connected to the rod. A pendant is connected to the wire and the substrate is either spot welded to the pendant or attached with Pt wires by means of holes drilled into the top of the substrate. The pendant provides about 30 g of force to push the substrate into the braze, while still having a flexible link to prevent damage to the load cell by inadvertent upward forces.

The substrate and crucible containing the braze material are contained within a quartz tube that can be evacuated or filled with inert gas. The Al_2O_3 crucible containing the braze is connected to a linear motor through a bellows coupling, enabling the crucible to be raised or lowered, while maintaining the environment in the tube. For tests run with the tube evacuated, the motor must be strong enough to overcome the atmospheric pressure acting

against the end of this evacuated tube (which is required when moving the crucible down at the end of the test). The rough pump and diffusion pump (with a cold trap) that was used was capable of achieving pressures as low as 5 X 10^{-7} torr, but generally evacuation was only continued long enough to achieve 1–2 X 10^{-6} torr at room temperature. The pressure rises on heating due to outgassing. To prevent high-temperature oxidation, the braze and substrate were preheated to about 300°C and outgassed by lowering the pressure to about 5 X 10^{-6} torr. Further heating was in stages maintaining the pressure below 10^{-5} torr. This was continued until the test temperature was reached, and the test was run with a vacuum of less than 10^{-5} torr. These are nominal pressures, which are measured in the vacuum system, between the furnace and pump. As such, they are somewhat lower than what is actually achieved at the heated substrate, but correspond to what might be measured in a typical vacuum furnace.

When an inert gas is used, an initial pump-purge procedure is used to remove the residual oxygen, and the gas is purified by a high-temperature getter prior to entering the test chamber. This getter is not used when a reducing gas is used.

A split, four-element elliptical radiant heater is used to heat the substrate and braze. The crucible acts as a susceptor, heating both the braze and substrate (whose initial position is within the crucible, but above the braze). A conventional resistance wound furnace can also

be used, but suffers from the limitation that a furnace temperature of more than 1000°C exceeds the quartz softening temperature, preventing the use of a resistance furnace at very elevated temperatures. The radiant furnace overcomes this problem because the quartz is largely transparent to the radiant energy and therefore does not heat to extremely high temperatures. The radiant furnace also heats and cools rapidly, thereby decreasing the time to perform an experiment. An Al_2O_3 tube could be used with a resistance furnace, but the lack of transparency makes setting up the test very difficult.

Three Pt thermocouples (TC) are used to control the temperature and to monitor the test. Two are located within the braze (protected by small-diameter closed-end Al_2O_3 tubes). The third TC is located within the crucible at the height of the substrate, but not within the braze. One of the submerged TCs is used to control the temperature; the other is used as a reference to increase the accuracy of the temperature measurements by averaging two readings and to get an idea of the temperature gradients that are present within the braze liquid. The temperatures of two thermocouples in the braze were within 1% (about 10°C) of each other (usually the difference was less than 5°C). These differences reflect the accuracy of the thermocouples (0.25% or 2.6°C) and gradients in the liquid, which cause spatial temperature variations.

The system is run under computer control. Velocity of the Al_2O_3 crucible is con-
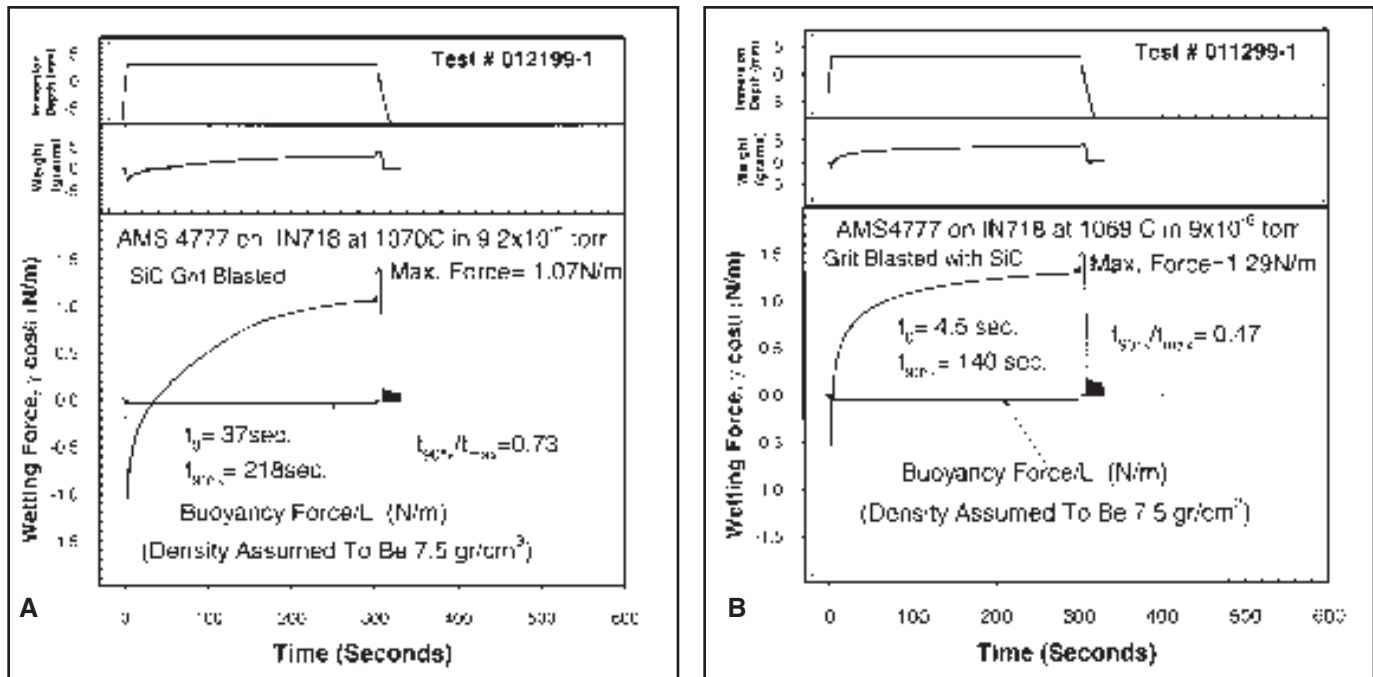
Fig. 6 — Wetting behavior of AMS4777 on IN718, which was grit blasted with SiC prior to wetting. A — At 1070°C in 9.2 ⟨ 10^{-6} torr; B — at 1069°C in 9 ⟨ 10^{-6} torr.

trolled, as is the depth of the substrate's immersion into the braze. The depth of immersion is determined by monitoring the weight of the system and by noting the reduction in weight associated with the braze contacting the substrate. This triggers a timing circuit, and the motion of the crucible is stopped at the time equal to the preset depth divided by the velocity of travel. The duration of exposure is also preset. When this time is reached, the crucible is lowered, completing the test. The computer creates a file containing output of the load cell, the temperature and position of the crucible, all as a function of time, in increments as small as 0.026 seconds. (The initial readings are at this high rate, but the rate is decreased after the first 30 seconds to one per second.) A strip chart recorder is also used to monitor the load cell and thermocouple signals as a function of time. This is done as a backup to the computer and to provide a real-time analog output.

Surface Tension and Wetting Angle Measurements

A wetting balance measures the wetting force per unit length, which is the product of the liquid surface tension and the cosine of the wetting angle. If the surface tension is known, then the wetting angle can be easily calculated. (Immersion devices have been built that measure the wetting angle (Refs. 4, 5), and wetting balances have been built that incorporate the ability to measure the wetting angle (Refs. 9, 10) in addition to the wetting force. These latter devices are, however, considerably more complicated than the one discussed here.) Surface tension values can be estimated from the literature (Refs. 32–38) or determined from Sessile Drop experiments (Refs. 10, 33, 39) performed on the exact braze being studied. Likewise, the braze density can be estimated from the literature (Refs. 32–38) or measured on the braze being studied.

The wetting balance can also be used to directly determine a lower limit to the surface tension. If there is initial nonwetting, a negative wetting force will be measured. If it is assumed that this is developed when the wetting angle is 180 deg ($\cos\theta = -1$), then a minimum value of the surface tension is obtained from this wetting force. This is a minimum value because the wetting angle is likely to be less than 180 deg, and the surface tension must be larger to yield the same value for $\gamma_{LV} \cos\theta$. Likewise, a minimum value for the surface tension can also be determined from the maximum positive wetting force, if it is assumed that this occurs when the wetting angle is reduced to zero ($\cos\theta = 1$).

Influence of Substrate Dissolution

As has been noted, wetting balances have been used for many years to study soldering. Their use to study brazing requires higher temperatures and control of the atmosphere. Both soldering and brazing involve reactive wetting, but with brazing there is the added complication that some of the surface being brazed may dissolve into the braze. (This can occur during soldering, but is much less prevalent because of the lower temperatures required for soldering.) Substrate dissolution complicates the wetting balance measurements in several ways. Dissolution of the substrate can alter the braze composition, but because the volume of the braze is orders of magnitude greater than the volume of the substrate that might dissolve, it is neglected here. (In the experiments described here, the volume of the braze was on the order of 5 cm³. The substrate being wet was 1.0 cm wide, 0.038 cm thick, and in all but one experiment, immersed 0.25 cm (in that test the immersion was only 0.025 cm). If all of the immersed volume dissolves, it is only about 10^{-2} cm³, so the effects of substrate dissolution on the braze composition were neglected.) In actual brazing, the influence of dissolved substrate cannot be neglected, since the volume of the braze is generally very small. This can be studied with a wetting balance if the braze composition is suitably altered to account for dissolved substrate. This was not done here because the focus was the relative effect of different grit blasting media.

In addition to possibly altering the braze composition, dissolution of the substrate can also alter the measured wetting force. This is best seen with reference to Equation 3, which describes the force measured by the load cell, relative to the weight before immersion into the braze. The actual force being measured is that
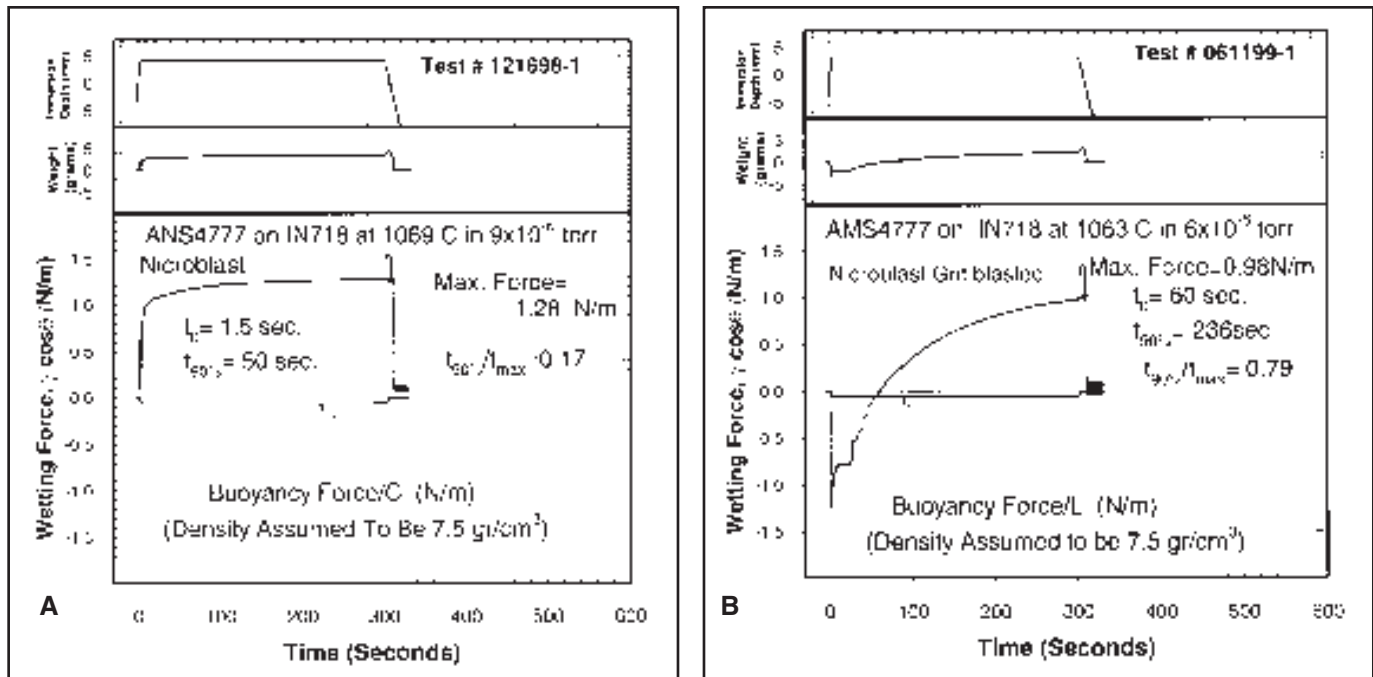
Fig. 7 — Wetting behavior of AMS4777 on IN718, which was grit blasted with Nicroblast prior to wetting. A — At 1069°C in 9 ⟨ 10⁻⁶ torr; B — at 1063°C in 6 ⟨ 10⁻⁶ torr.

given by Equation 3 plus the weight of the substrate, pendant, and support wire. When some of the substrate submerged into the braze dissolves, there will still be a wetting force exerted by the braze that wets the sides of the substrate above the submersion line. The only change in the force that is measured is that due to changes in the buoyancy force and weight of the dissolved substrate. If some, or all, of the substrate dissolves, then the buoyancy force associated with this material is decreased. If all of the material dissolves, then the $-\rho_b g V_d$ term of Equation 4 goes to zero, and the load cell will measure an increase in force, because of the decrease in the negative buoyancy force. If the volume of this dissolved material is V_d, then this increase is just $\rho_b g V_d$ (where ρ_b is the density of the liquid braze). The dissolution of a volume V_d of the substrate causes the substrate to get lighter by an amount $r_s g V_d$ (where r_s is the density of the solid substrate), i.e., the load cell will measure a decrease in force of $\rho_s g V_d$. Thus, some of the change in the net force that is attributed to the action of wetting may be due to the reduction in weight of the substrate. To correct for the decrease in the buoyancy, one must subtract $\rho_b g V_d$ from the measured force. To correct for the decrease in the weight of the dissolved substrate, one must add $\rho_s g V_d$ to the measured weight. The correction to F/L is thus

F/L correction for dissolution of
submerged substrate = $(\rho_s - \rho_b) g V_d / L$ (5)

Since V_d is generally small and $(\rho_s - \rho_b)$ is also generally small, this effect is small and can be neglected.

The preceding assumes that the only dissolution occurring is from material submerged in the liquid braze. Sometimes the braze that wets the substrate above the bulk braze causes dissolution through the thickness and along the sides of the substrate, or only a portion of the substrate is wet. In these cases, the perimeter is decreased, and it is necessary to correct the calculated wetting force for this change.

Experimental Procedures for the Illustrative Tests of Brazing on IN718

In these tests, the substrates were received after grit blasting. They were spot welded to the pendant, and then the substrate and pendant were degreased in alcohol. The pendant was then installed in the balance and the assembly pumped down and heated, as described above. The substrate being wet was 1.0 cm wide, 0.038

Table 1 — Braze Material and Blasting Conditions

Material	Grit Particle Size	Shape	Pressure	Standoff Distance
Al₂O₃	–240 (Mesh)	Angular	40 lb/in.²	3–4 in.
SiC	–240 (Mesh)	Angular	40 lb/in.²	3–4 in.
Nicroblast	–60 (Mesh)	Round	40 lb/in.²	3–4 in.

Table 2 — Nominal Composition of IN718 and AMS 4777 (AWS BNi-2) Wt-%

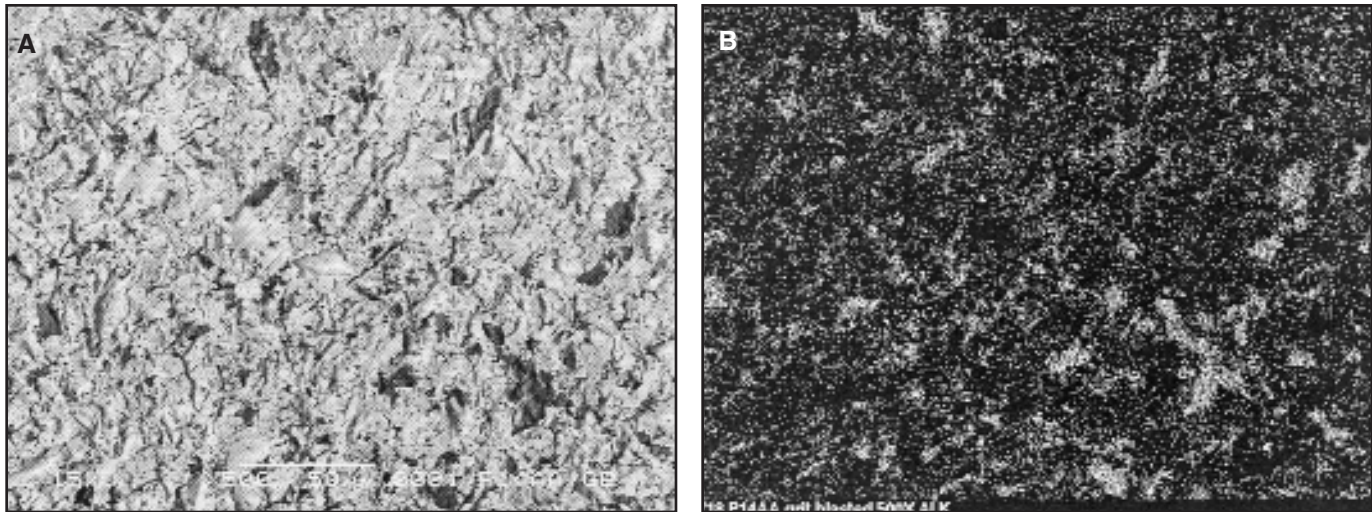|	Ni	Cr	Fe	B	Mo	Al	Ti	Si	S	P	C	Zr
IN718	Bal	19	18.5	—	3	0.5	0.9	—	—	—	0.08 Max	—
AMS 4777	Bal	7.0	3.0	3.1	—	0.05 Max	0.05 Max	4.5	0.02 Max	0.02 Max	0.06 Max	0.05 Max

Fig. 8 — A — IN718 grit blasted with Al_2O_3; B — Al dot map for the same area.

Table 3 — Wetting Test Results

Grit Blasted With	t_0 (seconds)	$t_{90\%}$ (seconds)	$t_{90\%}/t_{max}$	Max Force (N/m)
Al_2O_3	+600 (no wetting)	–(600 second test)	—	–0.18
Al_2O_3	367	569 (600 second test)	0.95	0.09
Al_2O_3	29	217	0.72	0.60
400μ Paper Polish after Al_2O_3	0.33 (shallow immersion)	132	0.44	1.25
400μ Paper Polish after Al_2O_3	6.4	120	0.40	0.99
SiC	0.06	21	0.07	1.24
SiC	4.5	140	0.35	1.29
SiC	37	218	0.73	1.07
SiC	113	273	0.92	0.48
Nicroblast	1.5	50	0.17	1.28
Nicroblast	1	41	0.14	1.25
Nicroblast	60	236	0.79	0.98

Except where noted, the duration of the brazing was 300 seconds at 1070°C.

cm thick, and immersed 0.25 cm or 0.025 cm in one test.

Three materials were used for the grit blasting: Al_2O_3, SiC, and Nicroblast (a proprietary Ni-Cr-Fe alloy powder made by the Wall Colmonoy Corp., which is easily wet by Ni base brazes). The grit size, shape, and blasting conditions are given in Table 1. The nominal compositions of the IN718 and AMS4777 are shown in Table 2.

Results and Discussion of the Influence of Surface Preparation on the Wetting of IN718

Figures 4–7 show the results of representative wetting balance tests run on IN718 substrates wet in vacuum with AMS4777 (AWS BNi-2) at 1060–1075°C. The tabulated values of the results for all of the tests are given in Table 3. These results are provided both as an illustration of

the use of the high-temperature wetting balance and to provide practical guidance on the preparation of surfaces for good wetting.

In Figs. 4–7, the top graph shows the immersion depth vs. time and shows when complete immersion started and stopped. Timing starts when the substrate first touches the braze. The zero depth denotes the depth where contact between the braze and substrate just begins (at time zero). In most experiments, the immersion depth was 0.25 cm. In one (Fig. 5), it was only 0.025 cm. Shallow immersions were primarily used in tests where the braze temperature was between the liquidus and solidus, so that the substrate does not hit the solid upon immersion. The test temperature used in the experiments shown here was above the liquidus, so this was not a problem. Thus, the deeper immersion depth was used in all

but one of the experiments (Fig. 5) described here. Negative depths denote where, at the beginning of the experiment, the braze has not yet contacted the substrate, or at the end of the experiment, where the braze has been pulled down away from the substrate. The very shallow immersion resulted in almost no buoyancy correction (which depends upon the volume of the immersed substrate) and a very short wetting time.

The middle part of each figure shows the weight change, relative to the initial weight, that is set to zero. The bottom graph is the most critical one. It shows the wetting force, as calculated from Equation 4. This force is the weight, measured by the load cell (zeroed with respect to the initial weight), plus the calculated buoyancy force ($\rho_b g V_d$), divided by the calculated perimeter, L (Equation 4). The buoyancy force divided by the substrate perimeter is also shown to illustrate that the buoyancy correction is small. (The buoyancy curve is shown for reference only; the buoyancy factor is already incorporated into the wetting force curve via Equation 4.) The immersion volume and perimeter have been corrected for the thermal expansion of IN718 (with a mean expansion coefficient (Ref. 40) of 10^{-5} cm/cm/F, so the correction is 1.02 for 1070°C (1958°F)). The correction to the volume is 1.04. (Only the specimen width and thickness have to be corrected for thermal expansion. The depth of immersion does not have to be corrected because it is set by the rate and time of immersion, not the specimen length.)

The buoyancy correction requires a knowledge of the density of the braze, which is unfortunately not a readily available quantity. For Ni (the primary constituent of the braze, which is about 80% wt-% Ni) at its melting point, the density
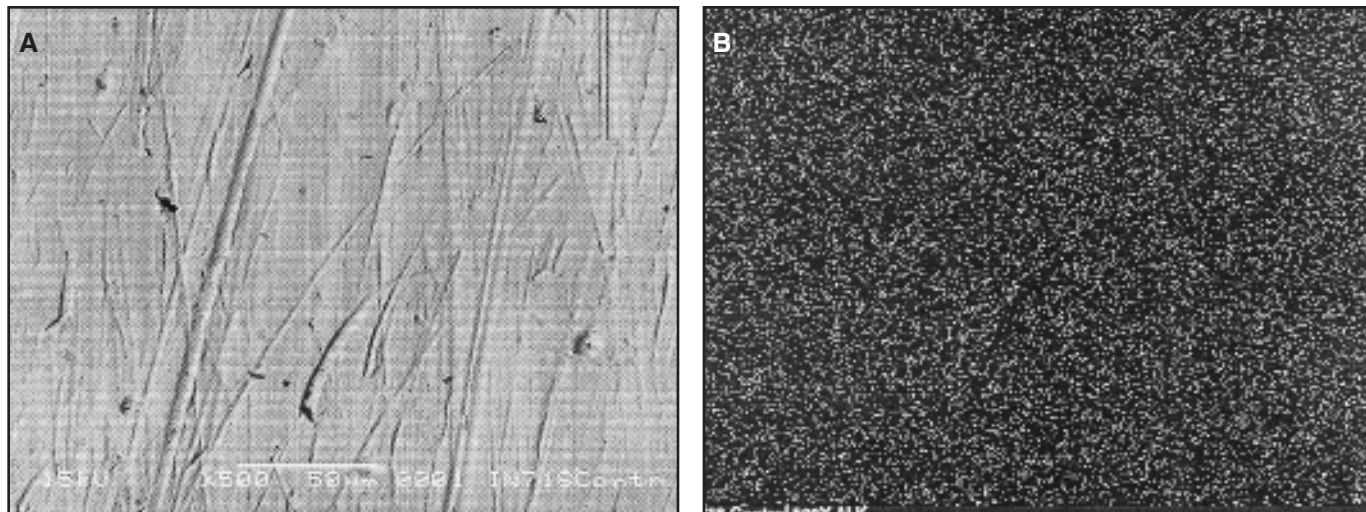
Fig. 9 — A — IN718 grit blasted with Al_2O_3 and then polished with 400-grit SiC paper to remove the alumina; B — Al dot map for the same area.

is 7.77 g/cm^3. Correcting for the lower melting point of the braze (because of the presence of the B and Si) raises the density to about 7.9 g/cm^3. This must be corrected for the other elements that are present (Table 2). Unfortunately, a complete data set does not exist for liquid density of all of the elements (that for B is not available) or the temperature dependence of the density (that for B and Cr are missing). Since the liquid densities of Cr, Fe, and Si are lower than that of Ni (especially the Si and most likely B), the density of the liquid braze is expected to be less than 7.9 g/cm^3. A value of 7.5 g/cm^3 was therefore employed for the calculation of the buoyancy term.

Fortunately, since the volume of immersed substrate is small, magnitude of the wetting force is not significantly dependent on the value of this buoyancy correction. The substrate being wet was about 1.0 cm wide, 0.038 cm thick, and in all but one experiment immersed 0.25 cm (in that test the immersion was only 0.025 cm). The immersed volume is thus only 9.5 X 10^{-3} cm^3 (9.9 X 10^{-3} cm^3 when the correction is made for the thermal expansion of IN718 [Ref. 40]). With a 0.25-cm immersion, the buoyancy term is about 0.05 N/m and only 0.005 N/m for a 0.025-cm immersion. If a density value of 7 g/cm^3 had been used, then the buoyancy correction would be reduced to about 0.047 for a 2.5-mm emersion and the wetting force would be reduced by only about 0.003 N/m, which is below the resolution of the measurements.

In most tests, there is an initial lack of wetting denoted by a negative wetting force. This negative force is not just due to the small buoyancy effect but is due primarily to the negative force that develops when the wetting angle is greater than 90 deg. When this is the case, the wetting

force acts to push the substrate out of the braze. When the wetting angle becomes less than 90 deg, a force is developed that pulls the substrate into the braze. The time to establish a positive wetting force, t_0, is listed on each figure and in Table 3. (Equation 4 incorporates a buoyancy term, so t_0 is not exactly the time where the net force measured by the balance becomes positive (downward).) In some tests (Table 3), wetting is almost instantaneous, occurring as the substrate is lowered into the braze. This gives t_0 values that are less than one second.

As the brazing exposure continues, the metallurgical reactions described in the introduction will continue, causing the wetting angle to decrease, thereby increasing the wetting force. The wetting force will approach the liquid braze surface tension as the wetting angle approaches zero.

The sharp increase and then subsequent decrease in the weight at the end of the test is associated with the lowering of the crucible, and resultant removal of the braze. Initially, due to the action of wetting, the downward motion of the braze pulls the substrate down, thereby increasing the measured weight. This is due to the inertial force associated with the downward acceleration of the crucible. As immersion is decreased further, the magnitude of this removal force is decreased, approaching zero when the braze is clear of the substrate. If the substrate is wet, then the final weight will be slightly greater due to the weight of braze that has wet the substrate, or reduced if some of the substrate dissolves in the braze. Since the calculated wetting force (shown in Figs. 4–7) includes an immersion-dependent buoyancy term, there is a small negative factitious contribution to the force

that develops when the braze is withdrawn and there is no buoyancy force actually acting on the substrate.

The wetting behavior can be quantified by the use of several figures of merit, which were evaluated and are shown in Table 3, and on the wetting figures. The maximum value of the wetting force determines the maximum capillary action. The higher the maximum wetting force and shorter the time for wetting, t_0, the better the wetting. These factors are, however, not the only ones that can be assessed. The shape of the wetting curve is also important. This is characterized by the time to reach 90% of the peak wetting force, $t_{90\%}$, and by this value divided by the duration of the test, t_{max}. The time to reach 90% of the maximum wetting force can be used to define the time required for brazing. When this time divided by the test time is small (i.e., 0.1 or less), it implies that the wetting curve is "rectangular," i.e., the wetting curve increases rapidly to close to its maximum value. When this ratio is close to one, it means that longer brazing times might have given a larger maximum wetting force.

The data of Table 3 reveal that there is a considerable amount of variability in the wetting balance measurements, and that grit blasting with Al_2O_3 generally yields the surface that is most poorly wet. This agrees with anecdotal experience. Figure 8 shows the Al_2O_3 grit blasted surface and a corresponding EDS dot map for aluminum. These figures show that Al_2O_3 grit blasting causes alumina particles to become embedded in the surface, and the wetting balance measurements show that it restricts wetting. Figure 9 shows the surface and an aluminum dot map of a substrate that was polished with 400-grit SiC paper after the alumina grit blasting. This
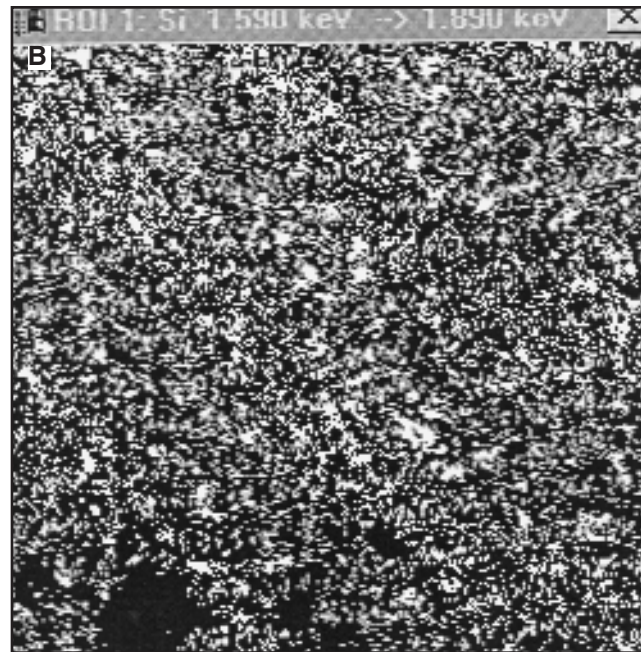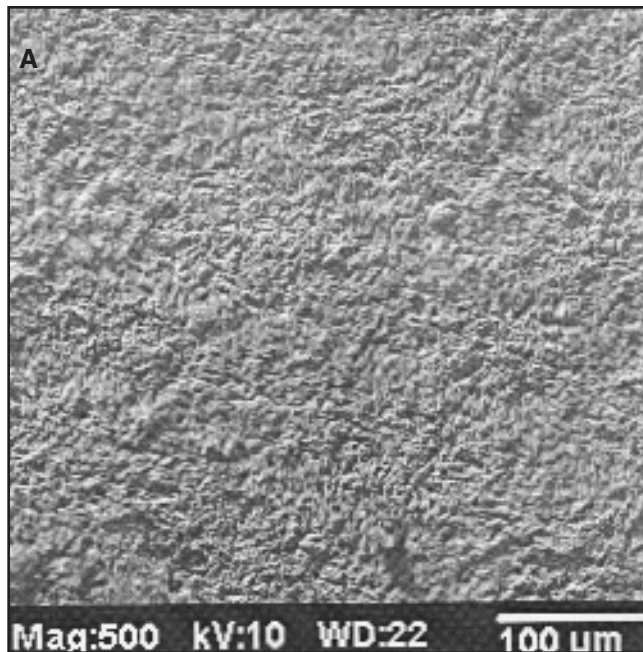
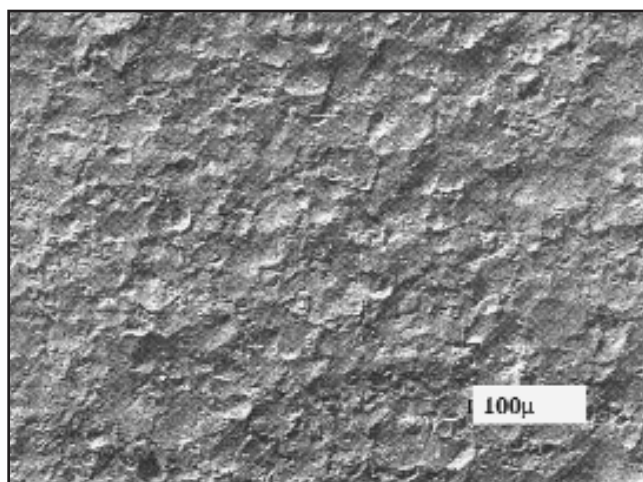*Fig. 10 — A — IN718 grit blasted with SiC; B — Si dot map for the same area.*

Fig. 11 — IN718 grit blasted with Nicroblast.

removes the Al_2O_3 particles. Aluminum is present in the background level of the alloy, but the large Al_2O_3 particles are absent. Figure 5 and Table 3 show that the removal of the Al_2O_3 greatly improves the wetting, both in terms of t_0 and the maximum wetting force.

Figure 10 shows the surface of a substrate grit blasted with SIC. This figure and Fig. 6 show that despite the presence of SiC particles embedded in the substrate, grit blasting with SiC generally produces a surface that is wet more easily than one grit blasted with Al_2O_3.

The best wetted surface was produced by the Nicroblast (media made by the Wall Colmonoy Corp.), which consists of –60 mesh Ni-Cr-Fe grit, that, as Fig. 11 shows, does not become embedded in the surface. This grit is larger and softer than the Al_2O_3 or SiC grit, and is made up of round rather than angular particles (Table 1). The Nicroblast grit being softer, larger and round, just indents the surface. Furthermore, the few particles that might be embedded are easily wet by the braze, because the Nicroblast is an alloy chemically formulated to be compatible with the brazes used for Ni-based alloys. The Nicroblasted surface gave low wetting times and high wetting forces, comparable to substrates that were mechanically polished.

While the degree to which the Al_2O_3 and SiC particles become embedded depends upon the exact nature of the grit and grit blasting conditions, it is clear that for the comparative conditions employed here, the Nicroblast produces a much better surface condition for subsequent brazing than Al_2O_3 and SiC. Presumably, other metal grit that does not become embedded and could be easily wet with the braze would give similar results.

Table 3 and Figures 4–7 clearly show that the method of surface preparation plays an important role in determining the degree of subsequent wetting. Al_2O_3 grit blasting is clearly detrimental and should be avoided, or if it must be used it should be followed by a mechanical polish to remove any embedded Al_2O_3 grit. SiC is less detrimental to subsequent wetting than Al_2O_3, but in at least one test it did retard the wetting time and reduced the wetting force. The Nicroblast produced a surface that was generally comparable to that observed with a mechanically polished surface. Wetting was accomplished in short times, with the wetting force reaching 90% of the maximum observed value in less than 5 minutes. Table 3 shows that with the most favorable surface preparation (mechanical polishing or grit blasting with Nicroblast), five minutes at the brazing temperature is sufficient to ensure good wetting. Extending the brazing time to ten minutes will increase the wetting force by only about 10%.

Conclusions

Technical advancements in the temperature and environmental control of a wetting balance have enabled this device to be used for the study of braze wetting. These advancements include the following:

1) The system has been operated at temperatures as high as 1225°C. (The radiant furnace is capable of 1450°C, but has only been successfully used to 1225°C.)

2) The tests can be run in vacuum (to about 10^{-5} torr at ~1070°C) and in inert gas.

3) A time resolution of 0.026 seconds was achieved.

4) If the surface tension of the braze is known (measured separately), then the wetting angle as well as the wetting force can be determined.

A study of the wetting of a Ni-based alloy (IN718) by AMS4777 (AWS BNi-2) on substrates grit blasted with different media (or mechanically polished to remove embedded particles) has shown the following:

1) Grit blasting of IN718 with Al_2O_3 retards wetting.

2) The Al_2O_3 becomes embedded in the substrate.

3) Mechanical polishing to remove the Al_2O_3 embedded in the surface restores good wetting.

4) Grit blasting with SiC produces a more easily wet surface than one grit blasted with Al_2O_3, even though SiC particles also become embedded in the surface.

5) Of the three grit blasting media that were considered, Nicroblast produced the most easily wet surface. Particles were not embedded in the surface. Its wetting was comparable to that observed when the substrate had been mechanically polished to remove embedded Al_2O_3.

References

1. Milner, D. R. 1958. *British Welding J.* (3): 90–105.

2. Lea, C. 1990/1991. *Soldering and Surface Mount Technology* (4): 8–13, (4): 14–22, (5) 46–55 (1990), (6): 4–9, (7): 10–13 (1991).

3. Humpston, G., and Jacobson, D. M. 1993. *Principles of Soldering and Brazing*, ASM International: Materials Park, Ohio.

4. Mayhew, A. J., and Wicks, G. R. 1971. Solderability and contact angle. *Proc Internepcon*, pp. 45–55.

5. Mayhew, A. J., and Monger, K. S. 1972. Solderability by meniscometry. *Proc Internepcon*, pp. 53–64.

6. Nakae, H., Yamaura, H., and Sinohara, T. 1989. *Materials Transactions*. JIM. 30:423–430.

7. Shipley, J. F. 1975. *Welding Journal* 54(7): 357-s to 362-s.

8. Naidich, Ju. V., and Chuvashov, Ju. N. 1983. *J. of Materials Science* 18: 2017–2080.

9. Rivollet, I., Chatain, D., and Eustanthopoulos, N. 1990. *J. of Materials Science* 25:, 3179–3185.

10. Eustopoulos, N., Nicholas, M. G., and Drevet, B. 1999. *Wettability at High Temperatures*. Amsterdam,The Netherlands: Pergamon Press.

11. Chappuis, J. *Multiphase Science and Technology*. Eds. G. F. Hewitt, J. M. Delhaye, and N. Zuber, Hemisphere Publishing Corporation. 1: 387

12. Young, T. 1805. *Phil. Trans. Roy. Soc*. London, U.K. 95:65.

13. Dupré, A. 1869. *Theorie Mecanique da la Chaleur*, Paris .

14. Gibbs, J. W. 1879. *Trans. Conn. Acad.* Vol 3, p. 343; also included in *Scientific Papers of J. W. Gibbs*, V1, N.Y.: Dover Publications, 1961, p. 314.

15. Bondi, A. 1953. *Chem. Rev.* 52: 417–458.

16. Johnson, R. E., Jr. 1959. *J. Phys. Chem.* 63:1655–1658.

17. Goodrich, F. C. 1969. Thermodynamics of fluid interfaces. *Surface and Colloid Science* Ed. E. Matijevic, Wiley-Interscience. Vol. 1, pp. 1–37.

18. Padday, J. P. 1969. Surface tension, part I — The theory of surface tension. *Surface and Colloid Science*. Ed. E. Matijevic, Wiley-Interscience. 39–99.

19. Johnson, R. E., Jr., and Dettre, R. H. 1969. Wettability and contact angles. *Surface and Colloid Science* Vol. 2. Ed. E. Matijevic, Wiley-Interscience, pp. 85–153.

20. Johnson, R. E., Jr., and Dettre, R. H. 1993. Wetting of low-energy surfaces. *Wettability*. Ed. J. C. Berg, Marcel Dekker, Inc. pp. 1–73.

21. Schwartz, M. M. 1987. *Brazing*. Materials Park, Ohio: ASM International.

22. Defay, R., and Prigogine I., with the collaboration of Bellemans, A. 1966. Translated by D. H. Everett, *Surface Tension and Adsorption*, New York, N.Y.: John Wiley and Sons, Inc.

23. Aksay, I. A., Hoge, C. E., and Pask, J. A. 1974. *J. of Physical Chemistry*, Vol. 78, pp. 1178–1183.

24. Pask, J. A. 1987. *Ceramic Bulletin*, 66: 1587–1592.

25. Yost, F. G. 1992. Fundamentals of wetting and spreading with emphasis on soldering. *The Metal Science of Joining*,. Ed. M. J. Cieslak, et al., The Minerals, Metals and Materials Society, pp. 49–59.

26. Yost, F. G., and Romig, A. D., Jr. 1988. *Mat. Res. Symp. Proc*. 108: 385–390.

27. Ambrose, J. C., Nicholas, M. C., and Stoneham, A. M. 1992. *Acta Met. Meter.* 40: 2483–2488.

28. Delannay, F., Froyen, L., and Deruyttere, A. 1987. *J. Materials Science* 22: 1–16.

29. Boettinger, W. J., Handwerker, C. A., and Kattner, U. R. Reactive wetting and intermetallic formation. *The Mechanics of Solder Alloy Wetting and Spreading*. Eds. F. G. Yost, F. M. Hosking, and D. R. Frear.

30. Orr, F. M., Jr., Scriven, L. E., and Chu, T. Y. 1977. *J. Colloidal and Interface Science* 60: 402–405.

31. Solomon, H. D., DeLair, R. E., and String, L. H. 2001. Self-Immersing Wetting Balance, U.S. Patent 6,286,368 B1.

32. Allen, B. C. 1972. Surface tension of liquid metals. *Liquid Metals — Chemistry and Physics*. Ed. S. Z. Beer. Marcel Dekker, Inc., pp. 161–212.

33. Iida, T., and Guthrie, R. I. L. 1988. *The Physical Properties of Liquid Metals.* Oxford Science Pub., p. 49.

34. Padday, J. P. 1969. Surface tension, Part 2. The measurement of surface tensions. *Surface and Colloid Science,* Vol. 1. Ed. E. Matijevic. Wiley-Interscience, pp. 101–251.

35. *Smithells Metals Reference Book*, 3rd Edition. 1992. Eds. E. A. Brandes and G. B. Brook, Butterworths.

36. Shiraishi, S. Y., and Ward, R. G. 1964. *Canadian Metallurgical Quarterly*, pp. 117–122.

37. Keene, J. 1993. *International Metals Reviews*, Vol. 38, pp. 157–192.

38. White, D. W. G. 1968. *Metallurgical Reviews*, No. 124, pp. 73–96.

39. Sundell, R. E., Solomon, H. D., and Correa, S. M. 1986. Minor element effects on gas tungsten arc (GTA) weld penetration — Weld pool physics. *Advances in Welding Science and Technology*, pp. 53–59. Materials Park, Ohio: ASM International.

40. Klopp, W. D. 1995. IN718. *Aerospace Structural Materials Handbook*, Code 4103, CINDA/USAF CRBA, Handbook Operation: Purdue University.