

Self-Propagating High-Temperature Synthesis as a Process for Joining Materials

Exothermic heat formation for certain compounds can be used for joining metals, with potential for dissimilar material combinations

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ABSTRACT. Self-propagating high-temperature synthesis or SHS is a process that allows production of a wide variety of ceramic and intermetallic compounds by highly exothermic reactions. It has been successfully used for simultaneous net-shape production, as well as joining, albeit the process of joining is in an embryonic state of understanding. In this study, the Gleebler® was employed for the systematic evaluation of the effects of heating rate (0.5° to 2°C/s), processing temperature (500° to 1300°C), hold time (2 to 60 min), and applied pressure (8.8 to 82.7 MPa), as well as minor variations of reactant composition, on the degree of reaction, joining integrity, and filler metal density and homogeneity in a model system of $3\text{Ni} + \text{Al} \rightarrow \text{Ni}_3\text{Al}$ reacted *in situ* between Alloy 600 end elements. For all temperatures above the reaction ignition temperature (T_i), where a liquid phase is formed (639.9°C), joining integrity was good. The degree of reaction, as well as product homogeneity and density, were all increased most by increased processing temperatures, with more modest effects from longer hold times and higher applied pressures. Heating rate had no apparent effect, although the range was narrow. Composition around the perfect stoichiometric ratio had a modest effect on density.

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Introduction

The Changing Challenges to Joining

The universe of materials once dominated by metals has grown to include advanced structural and technical ceramics and heat- and corrosion-resistant intermetallics and their composites, among others (Ref. 1). Metals have not been supplanted, just complemented and supplemented by these materials. But, metals too have changed. Metals, ceramics, intermetallics, and composites therefrom, are now "engineered" in every sense of the word, including: thoughtful formulation of composition from first principles of chemistry and physics; deliberate design of microstructure from first principles of materials science; highly controlled thermomechanical processing to develop that microstructure; and careful secondary processing into parts, devices, assemblies and structures that retain that microstructure and enable new levels of

performance, reliability, and economy (Ref. 2). One of the most important and challenging, but too often ignored, secondary processes that must frequently be accomplished is joining, and the joining of dissimilar combinations of metals, intermetallics, and ceramics is especially challenging.

Ironically, the very properties that lead to the selection of ceramics and intermetallics for use in assemblies, possibly with metals, in the first place also render them difficult to join. High melting temperatures, tendency to sublime or decompose rather than melt, intolerance to thermal shock, slow diffusion, lack of plasticity, and chemical inertness make fusion and nonfusion welding and brazing of ceramics difficult (Refs. 2, 3), while high melting temperatures, tendency to disorder after melting and resolidification, susceptibility to contamination by interstitial elements, and susceptibility to hot cracking pose similar problems for intermetallics (Ref. 4). Clearly, innovative methods for joining advanced ceramics and intermetallics to themselves, to one another, and to metals are badly needed (Ref. 5).

The Potential and Challenges of Joining Using SHS

A relatively recently identified process for synthesizing refractory inorganic ceramic and intermetallic compounds and alloys offers promise as a new means of joining these advanced materials. This process is self-propagating high-temperature synthesis (SHS) (Ref. 6), also known as combustion synthesis (Ref. 7) or reactive joining (Ref. 8). Based on its predominant mechanism of

Key Words

SHS Joining
Exothermic Reactions
Alloy 600
Metals
Dissimilar Materials
Process Parameters
Heating Rate
Hold Time
Applied Pressure
Processing Temperature

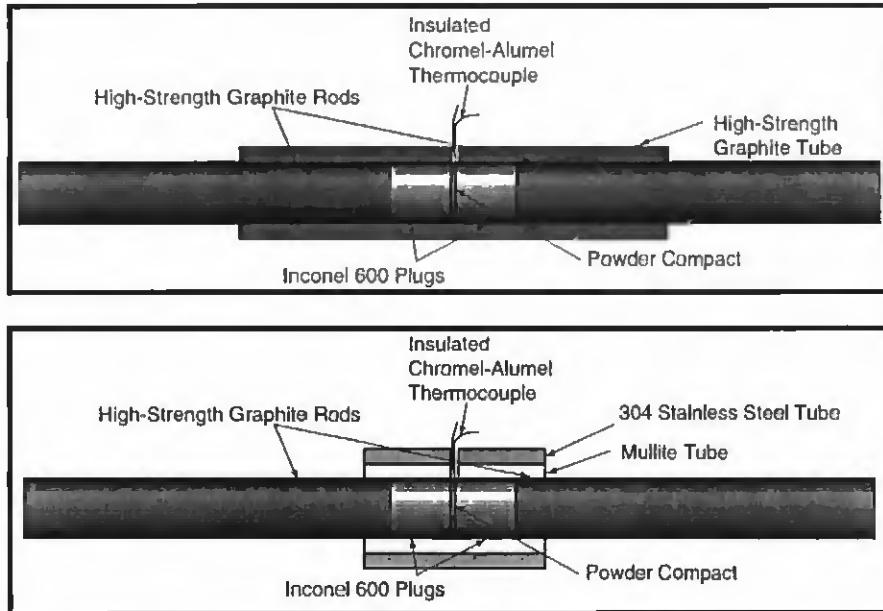


Fig. 1 — Schematic of the joint arrangement used in Gleble-based studies of SHS joining. A — Graphite containment tube and load-application plungers as well as the sandwiched, precompacted reactant powder and end elements; B — modified arrangement used for higher temperature, higher pressure joining conditions.

bonding, it falls under the AWS classification of exothermic brazing (Ref. 9).

In SHS, a reaction is initiated between reactants to form a compound for which there is a significant exothermic heat of formation. Reactants may be powdered solid elements, a powdered solid element and a gaseous element, or a powdered compound and an element or other compound, provided the result of reaction is a new compound with a higher heat of formation. Such reactions are generally referred to as "aluminothermic reactions," after the well-known reaction occurring between iron oxide (Fe_3O_{4s}) and pure aluminum (Al_s) to produce aluminum oxide (Al_2O_{3s}) and pure molten iron (Fe_l) in the Thermit™ welding process. Once it begins at a so-called triggering or ignition temperature (T_i), the reaction is sustained by the self-heating associated with exothermic compound formation. Adiabatic heating raises the temperature to a peak, known as the combustion temperature (T_c), as determined by an energy balance given by: $H_f = C_p \Delta T + H_m$, where H_f is the enthalpy of formation of the final compound (i.e., reaction product), ΔT is the temperature rise from T_i to T_c , C_p is the heat capacity of the various reaction components (including reactants, additives and diluents), and H_m is the appropriate value of melting enthalpy for every reactant and product (Refs. 10, 11). The peak temperature reached (or combustion temperature, T_c) depends on the particular combination of reactant and product melting events, with each melting

event consuming energy and lowering the peak temperature. Control of the peak temperature, through selection of reactant compositions, particle sizes, and precompacting density, is one of the primary means of controlling the process (Ref. 11).

In fact, the SHS process can be carried out in either of two distinct modes. In the "propagating mode," a small volume of reactants is heated locally to above T_i , and the reaction proceeds spontaneously along a fast-moving reaction front, leaving product behind. In the "simultaneous combustion mode," the entire reactant volume is heated above T_i , and the reaction occurs all at once. These different modes lead to different process challenges and different final product densities and cured-in stresses (Ref. 12).

A wide variety of materials has been produced by SHS including: oxides, nitrides, carbides, carbonitrides, hydrides, chalcogenides (e.g., sulfides, selenides), silicides, beryllides, aluminides and various intermetallic compounds and alloys (e.g., NiTi) (Refs. 6, 7). Besides synthesizing raw materials, the process has been successfully used for producing net shapes simultaneously with material synthesis in a process called "reactive sintering" (Refs. 13, 14). Here, the heat of reaction is used to produce a molten phase that leads to liquid-phase sintering. A natural extension of the process of reactive sintering is to joining (Refs. 8, 15–17). Here, the heat of compound formation can be used to accomplish joining, either simultaneous with substrate

synthesis and shaping (in a process of primary joining) or subsequently between pre-existing substrates (in a more familiar process of secondary joining). Actual atomic bond formation may be by liquid phase formation, wetting, and localized dissolution of the substrate surface (analogous to brazing), or by chemical reaction and/or interdiffusion in the solid state (analogous to solid-state reaction or diffusion brazing).

The process of SHS is attractive for joining ceramics and intermetallics, in monolithic or reinforced forms, to themselves, to one another, or to metals for the following reasons: 1) the required high temperatures needed for bond formation can be achieved efficiently through internal heat generation (vs. from external, power-consuming sources); 2) chemical compatibility with the substrates is relatively easy to achieve through proper reactant formulation; 3) the speed and highly localized nature of the reaction, occurring *in situ*, limits thermal degradation to substrates; 4) reinforcing particles, chopped fibers or whiskers can be readily incorporated into the product (i.e., joint filler metal) to enhance joint properties when joining composites; and 5) functionally gradient material (FGM) joints (Ref. 18) can be produced where the composition is varied across the joint to bridge chemical composition and physical and mechanical property incompatibilities and mismatches between dissimilar substrates.

Unfortunately, the SHS process for joining is in an embryonic state, is not well understood, and there are numerous problems to be overcome, including: 1) understanding the fundamental process and the role of various process parameters on joint quality, integrity and process kinetics; 2) understanding the role of and need for wetting by a liquid phase; 3) achieving dense joints, free of the porosity that accompanies most synthesis reactions due to retention of some residual interparticle voids, differences in specific volume between product(s) and reactants, or evolution of dissolved or adsorbed gases associated with the reactant powders; and 4) measuring and optimizing SHS joint properties.

Objective

This paper describes a study whose objective was to understand the role of the key parameters involved in joining by SHS, including temperature of processing, rate of heating to the processing temperature, hold time, applied pressure during processing, and reactant composition around the perfect stoichiometry. A

model system of $3\text{Ni} + \text{Al} > \text{Ni}_3\text{Al}$ reacted *in situ* between Alloy 600 nickel-based superalloy metal end elements was employed to avoid complications associated with the joining of dissimilar material types (e.g., ceramics to intermetallics or metals).

Experimental Procedure

A model system involving the reaction $3\text{Ni} + \text{Al} > \text{Ni}_3\text{Al}$ as the filler metal sandwiched between Alloy 600¹ metal end elements and reacted *in situ* was selected for initial study of process parameters to avoid complications with chemical incompatibility and mismatch of thermal coefficients of expansion associated with ceramic to metal or intermetallic joining (Refs. 19, 20).

Novamet INCO Nickel Type 123 3- μm powder was mixed for 30 min with the proper atomic proportions of Valimet Type H-15 15- μm aluminum powder using a WAB Impandex Turbula T2C mixer. Following mixing, 2-mm (0.08-in.) thick, 12.7-mm (0.5-in.) diameter disks were cold compacted from approximately 1-g quantities of powder at 345 MPa (50 ksi) using a Carver laboratory press and double-action die sets. These disks were sandwiched between 12.7-mm-long by 12.7-mm-diameter Alloy 600 cylinders machined from 12.7-mm-diameter rod stock. The sandwiched assembly was inserted to the midpoint of a high-strength graphite tube with 3-mm (0.12-in.) thick walls, and close-fitting high-strength graphite, solid cylindrical plungers were inserted into each end of the tube to permit load application and complete an electric path for resistance heating in a Model 1500 Gleeble®². A preprogrammed temperature-time cycle and simultaneous, but independently controlled, pressure-time cycle were applied by the Gleeble, using closed-loop feedback of specimen temperature from a 0.25-mm (0.01-in.) diameter Type K thermocouple embedded through the graphite containment tube wall at mid-span to the precompacted reactant disk. A schematic of the arrangement is shown in Fig. 1, while a photograph during an actual run is shown in Fig. 2.

Sandwiched joints were reacted in the simultaneous combustion mode by heating the entire assembly to a selected temperature at a controlled rate and holding at this temperature for selected times and under various pressures applied as soon as the target processing (and, usually, reaction) temperature was reached. Before full pressure was applied, a holding pressure of approximately 8.8 MPa or 1250 psi (corresponding to a minimum controllable clamping force of approxi-

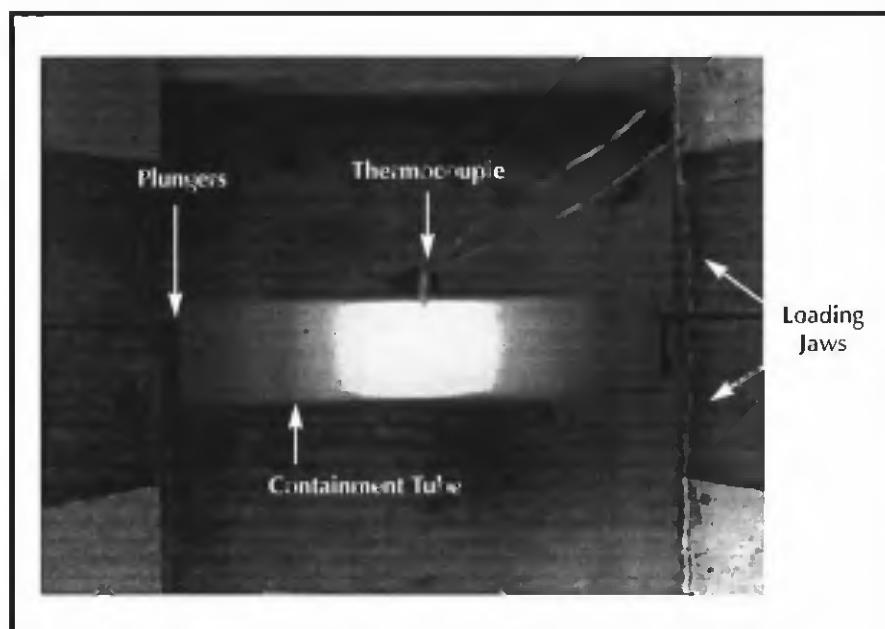


Fig. 2 — Photograph of an actual joint during its production by pressurized combustion synthesis or SHS. The cooler graphite load-application plungers can be seen exiting the glowing containment tube to be gripped by water-cooled copper loading jaws. A Type K thermocouple used for feedback control can be seen exiting the tube wall at midspan.

mately 250 lb) was applied. All testing was conducted in an argon-filled chamber available on this Gleeble for surrounding the test specimen, but still allowing heating, load application, data acquisition, and viewing through a Pyrex™ window.

Following processing, joint assemblies were removed from their graphite containment tubes and prepared for examination by optical and scanning electron microscopy (SEM) techniques. The presence and degree of reaction and bonding between *in situ* reacted filler metal and substrate, the density (or, conversely, the porosity content) of the filler metal, and the degree of reaction and homogeneity of the filler metal were all assessed. Porosity content was determined using computer-assisted quantitative metallographic techniques, while homogeneity was assessed using a combination of optical phase contrast and back-scattered electron element mapping.

Results and Discussion

Experimental Matrix

The experimental matrix of processing temperatures, heating rates, hold times, applied pressures, and reactant composition around the perfect 3:1 Ni:Al ratio for two heating rate/temperature/hold time/pressure conditions, is shown in Table 1. Processing temperatures were selected to range from below the known reaction ignition temperature

for Ni_3Al (nominally, 620°–630°C (1148°–1166°F), and corresponding closely to the temperature at which an Al-rich eutectic forms at 639.9°C/1183.8°F at 5.7 wt-% Ni) to progressively higher temperatures to avoid intermediate forms of Al-rich intermetallics, speed solid-state diffusion, and increase the amount of liquid phase present. Hold times were selected to range from immediate cool down following attainment of target processing temperature to 60 min to allow significant mass transport by diffusion and/or liquid infiltration to increase the degree of reaction, decrease the level of porosity, and increase the degree of homogeneity. Applied pressure was increased from the minimum achievable with the Gleeble (*i.e.*, 8.8 MPa) to progressively higher levels (limited by the breaking strength of the graphite containment tube) to assess the effect on product densification (*i.e.*, porosity reduction). Heating rate was varied from 0.5° to 2.0°C/s (0.9°–3.6°F/s being careful in these initial experiments not to overdrive the Gleeble) to observe any effects on reaction kinetics. Composition was adjusted from the ideal ratio to be rich in either Al or Ni, with atomic

1. The nominal composition of Alloy 600 is 76.0 Ni, 0.08 C, 0.5 Mn, 8.0 Fe, 0.008 S, 0.25 Cu and 15.5 Cr.

2. The Gleeble® thermal-mechanical simulator is produced by Dynamic Systems, Inc., (formerly Duffers Scientific Instruments) of Poestenkill, N.Y.

Table 1 — Matrix of Test Conditions and Resulting Porosity Levels in Joints Produced by SHS

Test No.	Temperature (°C)	Hold Time (min)	Pressure (MPa)	Heating Rate (°C/s)	Composition (at-%)	Porosity (vol-%)
0						27.7
1	500	2	8.8	1.0	Ni75Al25	14.3
2	500	15	8.8	1.0	Ni75Al25	22.3
3	500	60	8.8	1.0	Ni75Al25	— ^[a]
4	650	2	8.8	1.0	Ni75Al25	20.6
5	650	15	8.8	1.0	Ni75Al25	21.1
6	650	60	8.8	1.0	Ni75Al25	14.7
7	650	15	41.4	1.0	Ni75Al25	6.6
8	650	15	41.4	1.0	Ni65Al35	3.5
9	650	15	41.4	1.0	Ni80Al20	1.5
10	650	15	41.4	0.5	Ni75Al25	3.8
11	650	15	41.4	2.0	Ni75Al25	9.4
12	650	60	41.4	1.0	Ni75Al25	3.3
13	1000	2	8.8	1.0	Ni75Al25	7.0
14	1000	15	8.8	1.0	Ni75Al25	6.9
15	1000	60	8.8	1.0	Ni75Al25	2.1
16	1000	15	41.4	1.0	Ni75Al25	0.1
17	1000	15	41.4	1.0	Ni65Al35	0.1
18	1000	15	41.4	1.0	Ni80Al20	0.1
19	1000	15	41.4	0.5	Ni75Al25	0.0
20	1000	15	41.4	2.0	Ni75Al25	0.1
21	1000	60	41.4	1.0	Ni75Al25	0.1
22	1000	15	82.7	1.0	Ni75Al25	— ^[a]
23	1000	60	82.7	1.0	Ni75Al25	0.1
24	1300	15	8.8	1.0	Ni75Al25	0.1

(a) No measurement could be made.

3. A comprehensive report on the use, benefits, and shortcomings of the Gleebel for studying SHS joining is contained in T. Orling's Master of Science thesis entitled: Application of the Gleebel® to a Study of Fundamentals of the Self-Propagating High-Temperature Synthesis Joining Process, Rensselaer Polytechnic Institute, Troy, N.Y. June, 1994.

4. In fact, a lower concentration of oxygen (i.e., approximately 5 ppm) was possible with the pure argon atmosphere than with a vacuum achieved with a normal roughing pump.

percentages of 65Ni/35Al and 80Ni/20Al vs. 75Ni/25Al, respectively, to observe any effects on product form, density, or homogeneity.

Gleebel Performance

Without going into detail here³, the Gleebel offered an excellent, convenient means for systematically studying the role of process parameters for joining by SHS (or pressurized combustion synthesis as it is known when pressure is em-

ployed to densify the reaction product or filler metal). It enables temperature-time and pressure-time profiles to be accurately and simultaneously, but independently, varied and controlled. Although not done here because of fears that copious amounts of gas might be generated during the SHS reaction from interparticle void spaces or absorbed or adsorbed gases and contaminate the vacuum system, the Gleebel also permitted reaction atmospheres to be varied and controlled from vacuum, to inert gases, to reactive gas mixtures⁴. The Gleebel also offers the ready possibility of testing the elevated-temperature strength and thermal stability of joints, although this too has not yet been undertaken.

These advantages notwithstanding, there were several shortcomings to the Gleebel including: 1) the primary utility is for studying reactions in the simultaneous combustion mode, as the entire volume of reactant is heated (approximately) uniformly, although reactions taking place in the propagating mode are also of interest for joining; 2) elevation of the joint temperature upon reaction caused the Gleebel's closed-loop power control system to shut off (heating) current as soon as the reaction begins, possibly quenching the reaction; 3) monitoring of the reaction zone proper (in order to determine T_1 and T_c) requires additional instrumentation; and 4) utility of the Gleebel may be restricted to those materials that can be resistance heated, precluding study of many ceramics. Each of these shortcomings is being addressed through consultation with DSI technical staff, and most seem resolvable.

Initial concerns about contamination of the Gleebel's vacuum system by copi-

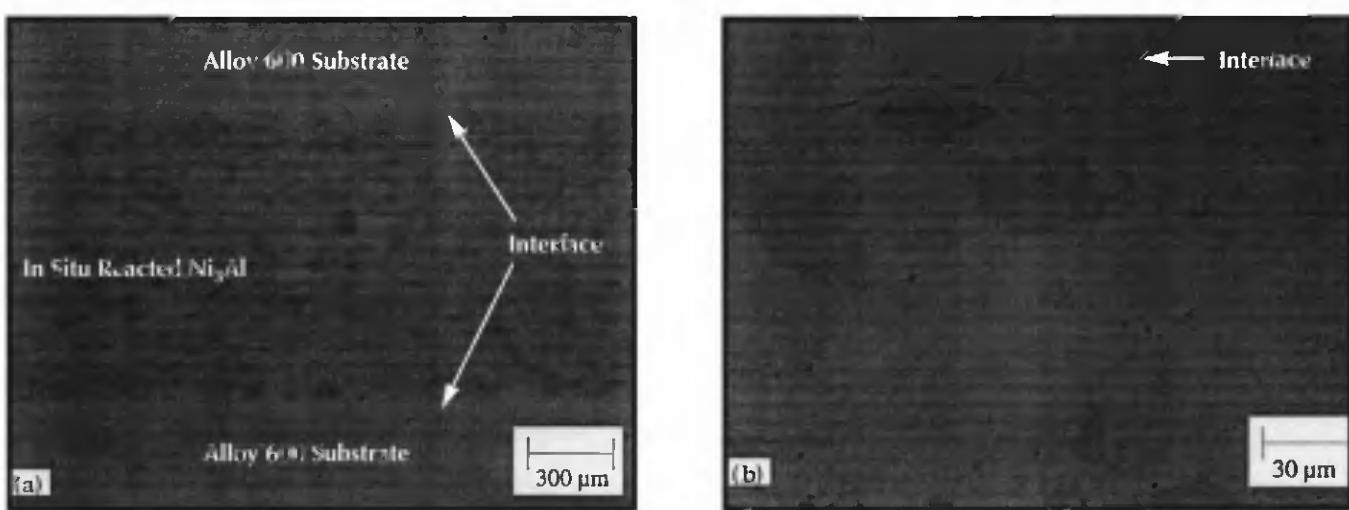


Fig. 3 — Typical joint interface between in situ reacted Ni_3Al and Alloy 600 end elements. A — Low-power magnification; B — high-power magnification. Joint (Specimen No. 21) was processed at 1000°C for 60 min under pressure of 41.4 MPa using a heating rate of 1°C/s. The interface is shown by an arrow.

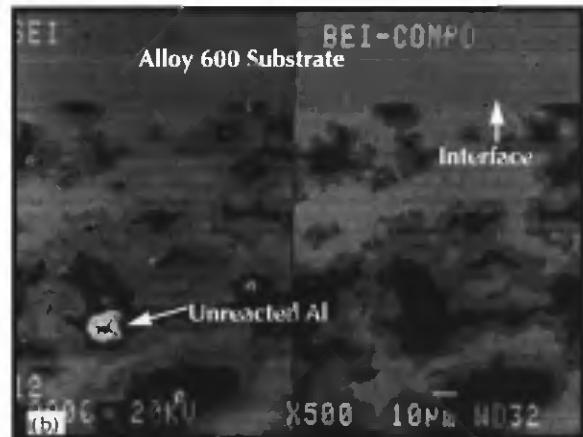
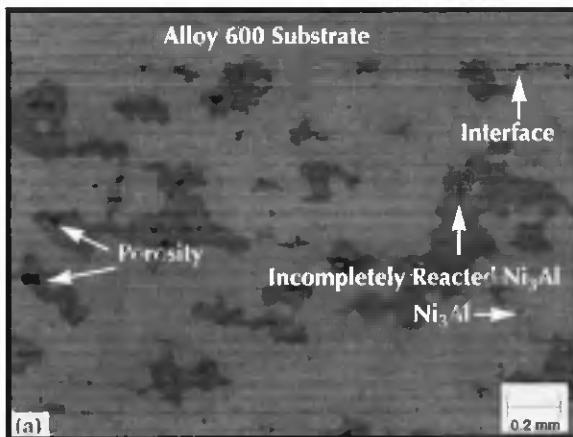


Fig. 4 — A and B Specimen (No. 12) processed at 650°C for 60 min under an applied pressure of 41.4 MPa. A — As-polished optical micrograph; B — secondary electron/back-scattered electron, split-screen scanning electron micrograph. Note the significant phase contrast indicative of incomplete reaction and product inhomogeneity. In A, black areas are voids where aluminum particles were originally; dark-gray areas (often surrounding voids) are Al-rich Ni₃Al; light-gray areas are Ni₃Al. In B, the only difference is that the very lightest areas are Ni-rich Ni₃Al.

ous amounts of gas were not warranted. No such evolution of significant quantities of gas was observed, probably because of the small volumes of reactant used in joining studies compared to earlier reactive sintering studies (Refs. 14, 21). As a result, future studies will include effects of atmosphere on atomic bonding mechanism and joint integrity, reaction kinetics, and product (*i.e.*, joint density).

Process Parameter Effects

For the matrix of process parameters evaluated (Table 1), bonding was achieved in every case where the processing temperature was above the point that some liquid phase was formed (*i.e.*, 639.9°C, for an Al-rich eutectic at 5.7 wt% Ni). This temperature corresponds closely to what has been reported as the

ignition temperature for Ni₃Al, *i.e.*, 620°–630°C (Ref. 22). It seems clear that some liquid phase formation is necessary to both cause and allow the reaction to proceed by gross heat and mass transport associated with the liquid, rather than just by diffusion.

While joint strength has not yet been measured, there did not appear to be any difference in joint integrity as processing temperature was increased from 650° to 1000° to 1300°C (1202°, 1832°, 2372°F). Bond interfaces, as shown in Fig. 3, were generally smooth, continuous, and free of any indications of contamination or excessive porosity compared to the bulk. Although some solid-state reaction was found to have occurred between Al and Ni particles at points of contact at 500°C (932°F), no such reaction was found at the interface with Alloy 600. Clearly, the rate of reac-

tion in the solid state at 500°C is far too slow to allow the reaction to proceed to completion in the times evaluated (*i.e.*, up to 60 min) and cause particle-to-particle bonding.

Increasing processing temperature above the ignition temperature had the most pronounced effect on degree of reaction, product homogeneity, and, especially, product or joint density. The effect on degree of reaction and product homogeneity can be seen in Figs. 4 and 5 for reaction temperatures of 650° and 1000°C for constant hold times of 60 min and constant applied pressures of 41.4 MPa (6 ksi). Optical and secondary and, especially, back-scattered scanning electron images revealed phase differences through contrast differences, which were later confirmed by elemental mapping, that were less pronounced as the processing temperature was increased. One

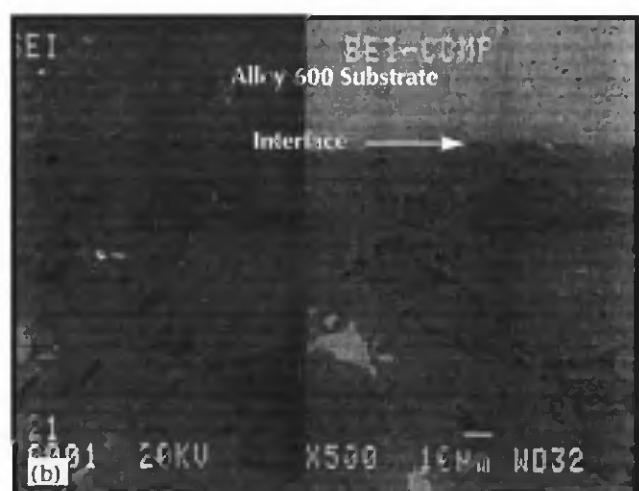


Fig. 5 — A specimen (No. 21) processed at 1000°C for 60 min under an applied pressure of 41.4 MPa. A — As-polished optical micrograph; B — secondary electron/back-scattered electron, split-screen scanning electron micrograph. Note far less phase contrast than seen at 650°C (in Fig. 4), indicative of more complete reaction and greater homogeneity of Ni₃Al.

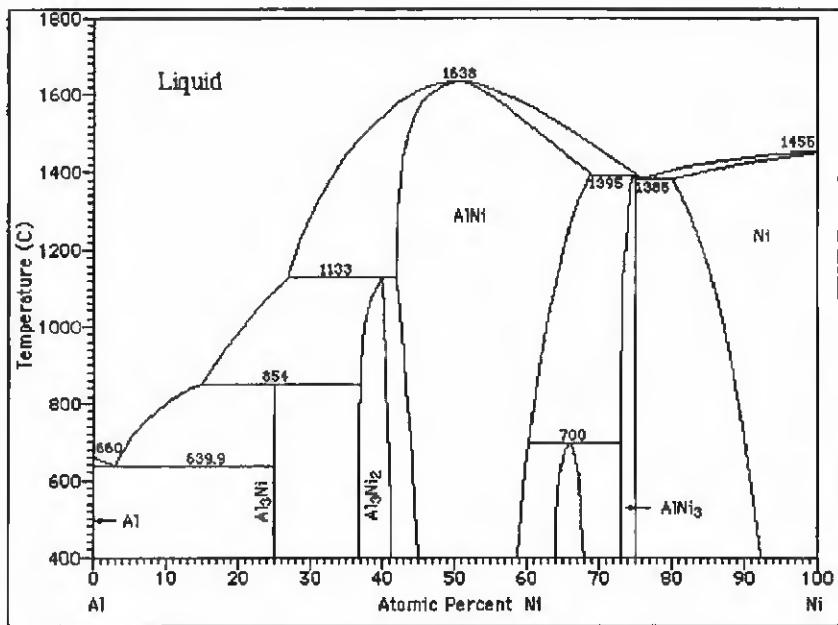


Fig. 6 — Binary phase diagram for Ni-Al, showing fewer Al-rich intermediate intermetallics before reaching Ni₃Al at higher temperatures.

reason for greater reaction and homogeneity is faster diffusion in the solid state, but other, more significant, reasons are greater proportions of liquid phase that facilitate both heat and mass transport, and occurrence of fewer intermediate forms of Al-rich intermetallic compounds between pure Al and Ni₃Al in the Ni-Al binary phase diagram as temperature is increased — Fig. 6. Close inspection reveals that Al is diffusing into the Ni more rapidly than Ni is diffusing into the Al, leaving Kirkendall voids where Al particles previously existed. Variation in composition can actually be tracked from pure Al to Ni₃Al through all inter-

mediate intermetallic compounds at any particular processing temperature.

The benefit of and practical need for higher temperature soaking to fully homogenize the product (joint) can be seen from Fig. 7. Here, the beneficial effect on homogeneity of exposure to 1200°C (2192°F) for 60 min after processing at 1000°C for 60 min, can be seen. Such treatments seem necessary for assuring that the reaction has gone to completion, and to homogenize and stabilize the reaction product or joint. Obviously, post-process homogenization should be performed at a temperature enough above planned use temperatures to assure phase

stability based on diffusion kinetics.

The effect of higher processing temperature on joint porosity content, or, contrarily, joint density, is very pronounced. This can be seen in Fig. 8, where the volume of porosity dropped from approximately 22% after processing at 650°C to 7% after processing at 1000°C to less than 0.1% after processing at 1300°C, all for constant hold times of 15 min and applied pressures of 8.8 MPa. The principal effect of temperature appears to be the formation of larger proportions of liquid phase that dramatically speed the transport of both heat and mass by capillary flow within the interconnected interparticle network.

The effects of hold time and applied pressure were far less dramatic than the effect of processing temperature, but were present. Increasing hold time following attainment of processing temperature increased the degree of reaction and product homogeneity far less effectively than increasing the temperature, as would be expected for diffusion-controlled, Arrhenius-rate processes. Porosity content was also reduced by increased hold time, with more pronounced effects at higher processing temperatures. This can be seen in Table 1 by comparing the porosity contents of Tests 4–6 (at 650°C and 8.8 MPa) to Tests 13–15 (at 1000°C and 8.8 MPa).

Increasing applied pressure, as might be expected, reduced porosity at any particular processing temperature for any particular hold time. This can be seen in Table 1 by comparing Tests 6 and 12 (for processing at 650°C for 60 min under pressures of 8.8 and 41.4 MPa) and, to a lesser extent, by comparing Tests 15, 21 and 23 (for processing at 1000°C for 60 min under pressures of 8.8, 41.4 and

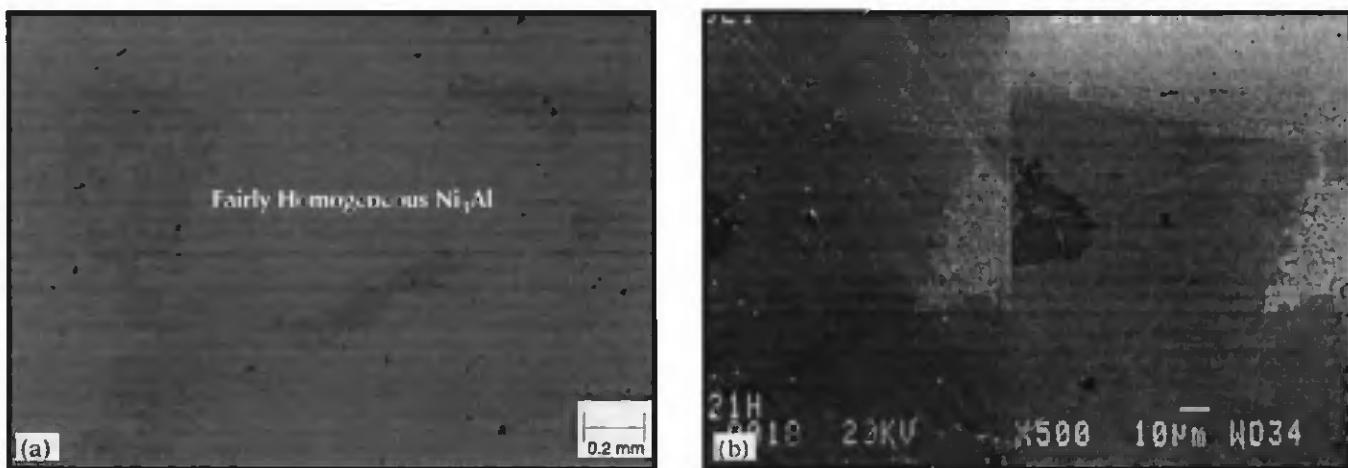


Fig. 7 — A specimen subjected to a postprocess homogenization treatment at 1200°C for 60 min after joining at 1000°C for 60 min reveals dramatically improved homogeneity as indicated by reduced phase contrast. A — As-polished optical micrograph; B — secondary electron/back-scattered electron, split-screen scanning electron micrograph.

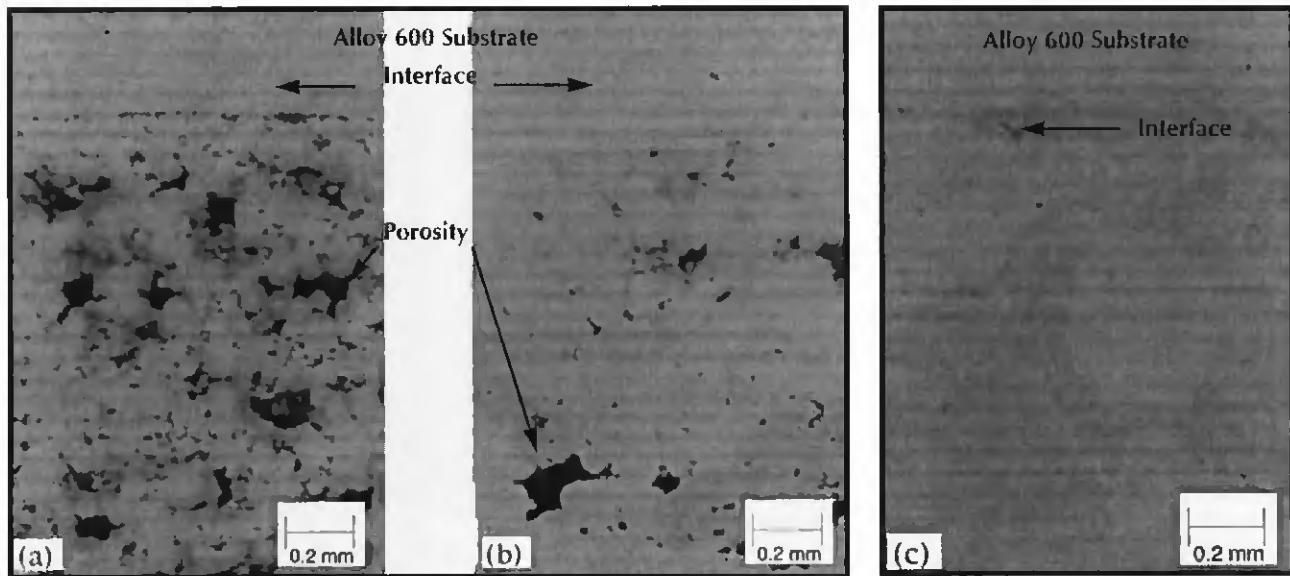


Fig. 8 — The effect of increased processing temperature on final product density. A — Volume percentage of porosity dropping from 22% after processing Specimen No. 5 at 650°C; B — to 7% after processing Specimen No. 14 at 1000°C; C — to less than 0.1% after processing Specimen No. 24 at 1300°C. All are for constant hold times of 15 min and applied pressures of 8.8 MPa. Porosity appears black in A and B.

82.7 MPa). Once the temperature of processing is high enough to produce significant liquid phase, the effects of increasing pressure is seen to be less significant. There is, thus, a practical limit to the level of applied pressure that is required and beneficial during joining.

No conclusive effects of increasing heating rate were observed over the range from 0.5 to 2°C/s. It can be expected that higher heating rates will influence the ignition temperature (T_i) slightly, and the peak or combustion temperature (T_c) a great deal (Ref. 11). Since a higher peak temperature will increase the amount of liquid phase present, process kinetics, especially for densification, can be expected to change (*i.e.*, increase) significantly. A higher heating rate has also been reported to decrease the amount of intermediate phase formed, in accordance with the elevated peak temperature (Ref. 23).

The special tests of off-stoichiometric mixtures of Al and Ni reacted at both 650°C and 1000°C for 15 min under a pressure of 41.4 MPa showed only minor effects. Perhaps most interesting is the observation among Tests 7–9 that the level of porosity in the product (joint) is reduced for otherwise constant conditions as the amount of either the Al or Ni is increased. The effect of increasing Al is undoubtedly to increase the amount of liquid present, thereby aiding densification by liquid transport. The effect of increasing Ni is to dilute the reaction and reduce the peak, combustion temperature, thereby altering the amount of liquid phase present. Although not explained, this effect on porosity has been

observed by others (Ref. 10).

Conclusions

1) The Gleeble® thermal-mechanical simulator is a valuable and innovative tool to studying joining by pressurized SHS, enabling simultaneous but independent control of temperature, applied pressure, and time, all of which are critical. Minor shortcomings appear to be solvable by refining experimental techniques, adding diagnostic instrumentation, and/or making minor changes to the control system.

2) Among the processing parameters of temperature, hold time at temperature, rate of heating, and magnitude of pressure applied during reaction, processing temperature is definitely most important as it increases the degree of reaction, decreases porosity, and increases product homogeneity as it is increased. Bonding seems to occur as long as the processing temperature is greater than the ignition temperature T_i for the particular SHS reaction involved. At least for this system, the presence of some liquid seems necessary for bonding. While SHS reactions can occur without a liquid phase being present, *i.e.*, in the solid state, the rate of reaction is often slow because material transport is by diffusion only. When a liquid phase is present, the transport of both heat (*i.e.*, temperature) and material is much faster.

3) Increasing the hold time at processing temperature leads to increased degree of reaction, reduced porosity, and increased homogeneity, but less effectively than increasing the temperature.

4) Applied pressure has a definite and pronounced effect on product densification, until processing temperature is high enough to produce a substantial amount of liquid. Liquid transport is far more effective than solid-state creep and diffusion at causing densification.

5) The rate of heating to the target processing temperature had no noticeable effect over the relatively narrow range from 0.5 to 2°C/s.

6) Reactant composition around perfect stoichiometry has an effect on product density, especially at lower processing temperature. The mechanism remains to be identified.

Acknowledgments

The work reported here was initially made possible by an Engineering Research Initiation Grant (ERIG) awarded to Dr. Messler by The Engineering Foundation and The Welding Research Council, with subsequent support from Lockheed-Martin Marietta's Knolls Atomic Power Laboratory.

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