Mokume Gane Firing Methods And Their Effects On Appearances And Bond Strengths

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Abstract

Mokume gane (wood-grained metal) is an ancient Japanese metalworking technique that involves the diffusion bonding of pure metals and alloy sheets into a billet that is forged, rolled and patterned, resulting in patterned materials. The practitioners of the art of mokume gane have found that billets that may look well bonded initially can fail catastrophically during the reductions which create the patterned materials.

In 2005 James Binnion, Andrew Nyce and Stewart Grice studied solid-state diffusion-bond strength as a function of open and closed torque plate bonding apparatus, utilizing the newly developed Thermal Expansion Mismatch Torque Plate System (TEMTP). Their paper (presented at the 2005 Santa Fe Symposium) demonstrated that the TEMTP system resulted in increased bond strength compared to the open torque plate system.[1] However, the authors did not include downstream processing within the scope of their study nor did they consider the effects of time and temperature on bond strength. In 2009, Ploof, Grice and Nyce presented a paper that looked at the role reduction plays in developing bond strength.[2] Several styles of reduction were examined with various results.

This paper is an adjunct to both of these preceding papers. This paper examines a method of liquid-phase bonding and the resulting mokume gane's strengths and appearances. It includes a "how to" based on many years of trial and error, and process improvements, which will enable someone with little mokume gane experience to make 'torch-fired' billets in a reasonably well-equipped studio, and to begin to use mokume gane in their own designs.

Introduction

Making mokume gane in the small shop using solid-state diffusion poses many technical problems and makes ownership of several expensive tools desirable. Typically, billets fired using this method yield excellent quality mokume gane if fired and reduced properly (please see Santa Fe Symposium papers 2005 & 2009),[1,2] and enough material to make several rings. Scale is limited mainly by size of the kiln, torque plate size, and by the method of reduction to usable stock. Methods employed for reduction typically include hydraulic pressing (both hot and cold, although press size severely limits the size of billets that may be cold pressed), hand forging and cold rolling (the limiting factor here is the maximum opening of the mill, which is typically 6-12mm for mills common to the small jeweler's shop). Billets are typically in the size range of 38mm x 25mm (1.5″ x 1″) for ease of processing. The cost of tools as well as the cost of materials to fire a billet of that size prevents many from attempting to make mokume gane.

Steve Midgett, author of *Mokume Gane, A Comprehensive Study*, describes construction and use of a mini-kiln for firing small billets of mokume gane in the range of 13mm x 25mm (½″ x 1″).[3] This employs a simpler method, liquid-phase diffusion bonding, working on a smaller scale. This kiln is constructed of a heavy-duty C-clamp centered in a round firing chamber. Two fire bricks are used, with one-half of the firing chamber carved in each. A small port is left for a hoke torch tip with another small hole acting as a viewing port. Many who have made this kiln, the author included, have had difficulties bonding mokume billets when using it. Complaints include the inability to accurately see the flame to properly adjust fuel/oxygen ratios, as well as an inability to see the 'flash' of the layers through the small viewing port as they reach the liquid phase. But this method does allow for many to attempt to make mokume gane without investing in expensive tooling and other equipment.

Several years ago, after hearing about a few artists exploring liquid-phase diffusion bonds, another artist and I decided to explore this method of manufacture of mokume gane. We attempted several methods, from the construction of larger kilns to other types of containment vessels to hold heat and contain the sheets of materials we were attempting to bond. A method was devised that allowed for firing of small billets. We also discovered that these billets seemed to exhibit great strength. We were able to manipulate these billets far more aggressively than those bonded by solid-state diffusion. The differences in strength from the liquid-phase to the solid-state phase billets were remarkable. Typically, reductions using a hammer and anvil could be taken in the neighborhood of 50-60% without heating or annealing steps, and the material could even be placed with the layers perpendicular to the forging surface and aggressively forged without breaking the bonds.

This was a fabulous observation, as one of the most difficult things to do is reduce the well-bonded solid-state billet. It is very easy to cause failures by working too aggressively. One of the key things in a manufacturing environment is discovering anything that saves time and still results in a quality product. We observed that these billets were fast and easy to make and quite strong. Both of these traits could speed up the process of manufacturing mokume gane.

Unfortunately, size did seem to be the limiting factor, with billets difficult to fire when larger than one square inch and greater than one-half inch thick. When this method was mentioned and touted as an option to several well-established makers of mokume gane, it was dismissed as making mokume gane that looked 'muddy' compared to solid-state diffusion-bonded billets.

This paper details the torch-firing method best practices as developed over the past several years, examines the strength of liquid-phase billets compared to solid-state billets using best practices as developed during the 2009 paper ("Mokume Gane Billet Reductions and Their Effects on Bond Strength"[2]), and compares appearances of the two methods to determine if materials look' muddier' when fired one way versus another.

Experimental Objectives

- Detail a method of 'torch-fired' liquid-phase bonding of mokume billets.
- Determine relative strengths of liquid-phase billets versus those fired using solid-state diffusion.
- Compare the appearance of patterned materials made from billets fired using both methods.

A series of experiments were constructed in order to meet our objectives. A total of six billets were liquid-phase bonded, and one billet was solid-state bonded.

- Four of the six liquid-phase billets were used to test the strength of the bonded materials against data collected in last year's paper.
- One of the six was to be used to make patterned sheet for appearance comparisons.
- The remaining billet was used for micrography to compare diffusion-zone sizes in the as-fired state prior to any reductions.
- The solid-state billet was for comparison to the above billet's diffusion zones and for manufacture into patterned sheet for appearance comparisons.

Billet Materials

All billets used for bond-strength testing and appearance comparison were 14 karat palladium white gold (14K Pd W) and sterling silver (.925), due to the author's observation of the ease of firing this particular metal combination and for comparison purposes to last year's bond strength testing. The Vickers hardness of the 14K Pd W and .925 are fairly close compared to some other metal combinations, the particular color (gray and white) of the combination is currently very popular in the consumer market, and the bond is very easy to manipulate in the studio without much risk of failure compared to some other combinations.

It is important to state that all research presented is based around the above material combination. Much testing and comparison of notes has been done between many makers of mokume gane, and all data point to the fact that every unique combination of materials has its own unique behavior when manufactured into mokume gane materials.

Stacking Order

Liquid-phase billets were comprised of 15 alternating layers of 14 karat palladium white gold and sterling silver measuring 38mm x 13mm x 20g (1.5″ x .5″ x 20g) with the outermost layers being 14 karat palladium white gold. The solid-state billet was comprised of 15 alternating layers of 14 karat palladium white gold and sterling silver measuring 25mm x 38mm x 20g (1″ x 1.5″ x 20g) with outermost layers of white gold.

It is important to note that the metal with the lowest liquidus should not be used for the outermost layers of torch-fired billets. This will prevent the outermost layers from sticking to the stainless steel binding wire required to hold the billet together.

Equipment Used

- Liquid-phase billets were fired using a Smith acetylene and ambient air torch, with a #4 torch tip (3/8″ opening).
- 24 gauge stainless steel binding wire.
- Superior Flux No. 6.

The solid-state billet was fired with the equipment detailed in the 2009 paper.

- Hydraulic press was the 55-ton H frame style used in many studio metalsmith shops.
- Rolling mills were the Durston powered rolling mill, over and under style, one with a maximum opening of 6mm and the other with a maximum opening of 12mm.
- 150-pound blacksmith's anvil, 2.5-pound cross peen hammer, 2″ x 2″ blacksmith's flatter, and 8-pound sledge hammer.
- Various hand tools common in the well-equipped studio.
- Photo micrography provided by Hoover and Strong.
- In-studio photography using a USB camera.

Billet Firing Procedure

All sheet to be liquid-phase diffusion bonded into billets was inspected visually and cleaned using a mounted 3″ buff (Figure 1).

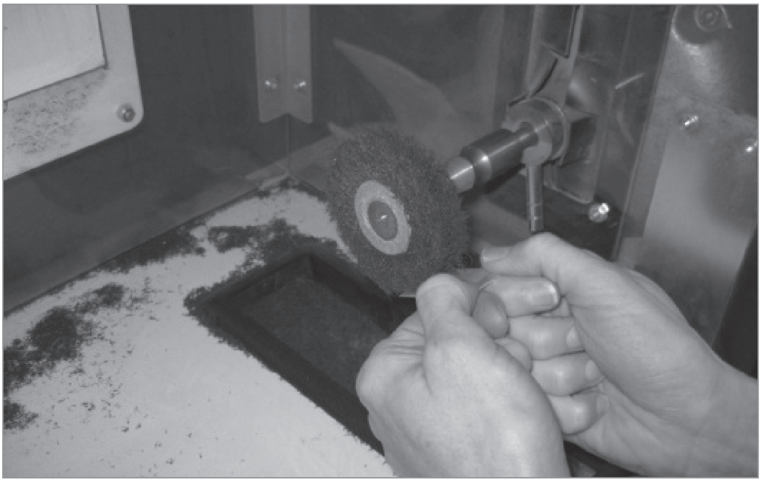

***Figure 1** After cleaning, the metal was stacked into its alternating layer sequence. This stack had little contact between layers due to twisting and deformation caused by shearing the metal into size for firing of the billet (Figure 2).*

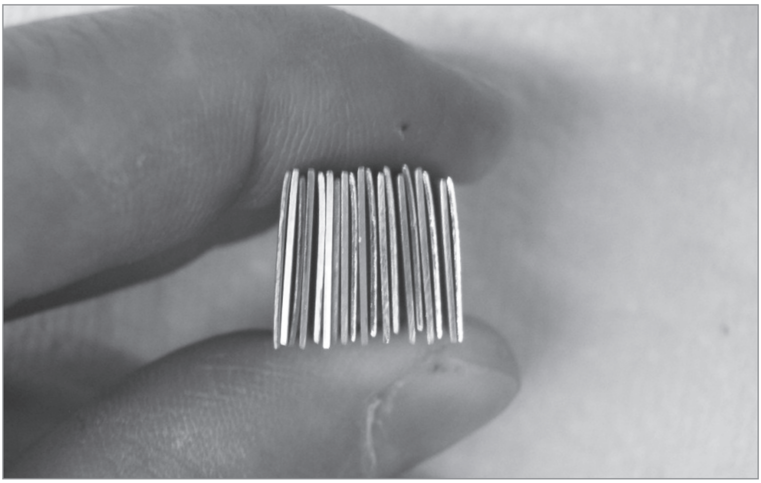

***Figure 2** The stacked sheets were pressed between two metal blocks to 10 tons of pressure. The purpose of this pressing is to flatten the sheets and increase contact prior to wrapping in the prepared stainless steel binding wire (Figure 3).*

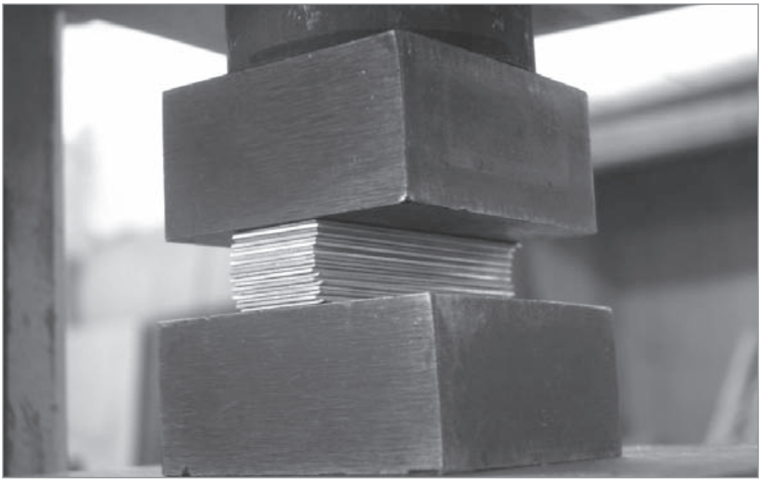

Figure 3 *After pressings, billets were secured with twisted stainless steel binding wire (Figure 4).*

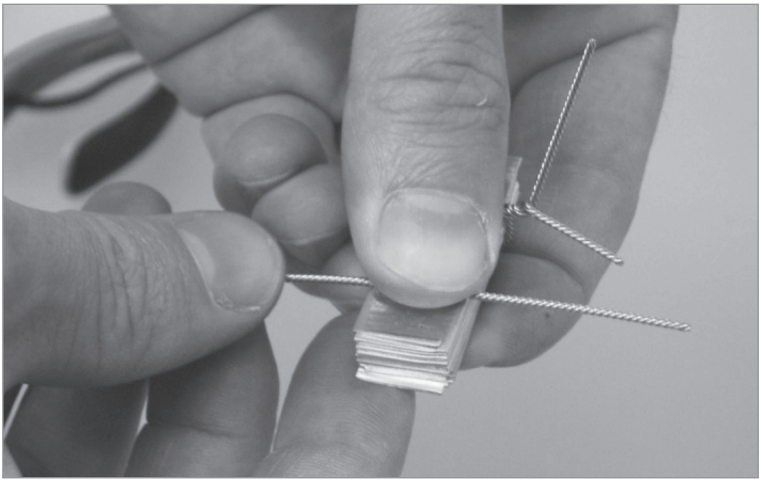

Figure 4 *The binding wire was prepared as follows: a wire of approximately six feet in length was folded in half twice and then twisted using a rotary tool (Figures 5-9).*

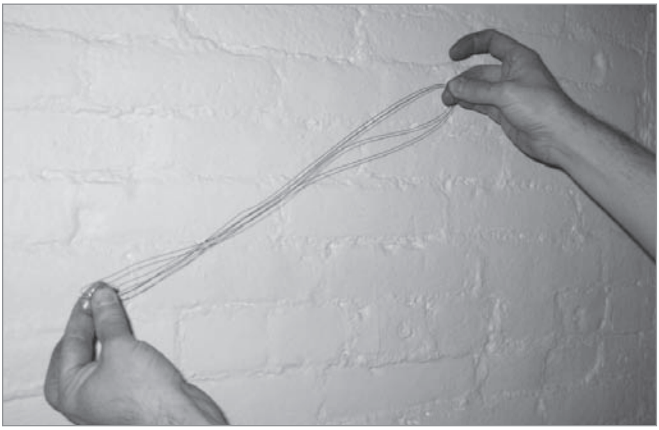

Figure 5

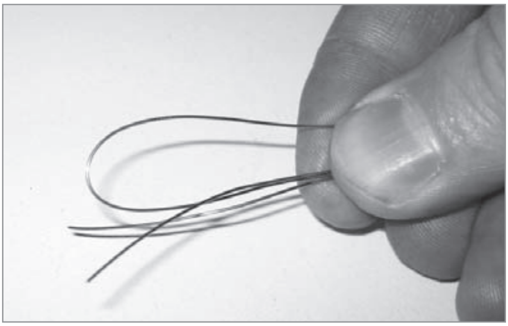

Figure 6

Figure 7

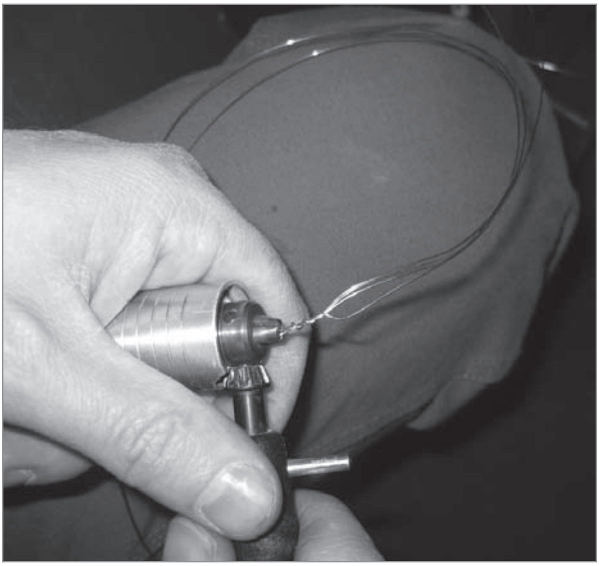

Figure 8

***Figure 9** The wire-bound billet was then heavily fluxed and placed on a fire brick in a rotating pumice pan (Figure 10).*

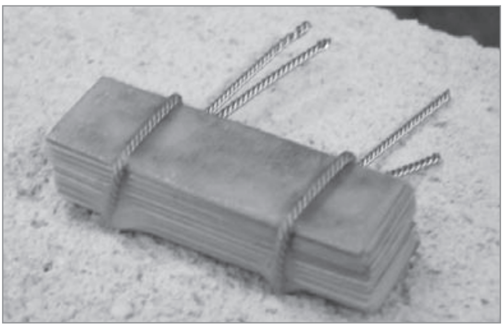

***Figure 10** The Smith air acetylene torch equipped with tip #4 was lit and adjusted to about 65 -70% of maximum output. Torch air intake holes were partially obscured with the hand to create a more reducing atmosphere (Figure 11).*

Figure 11

The billet was then evenly heated from both sides. Firing follows these stages: First the liquid in the flux boils off, leaving a crusty, white surface; then the flux turns clear and exhibits a lot of surface tension; finally the flux flows freely and the billet starts to glow if viewed in a darkened soldering area. The time that this process takes is dependent on the size of the billet and torch, but it generally takes about 75% of the total time spent to fire the billet.

Billets fired in the liquid-phase 'torch fired' method are judged by eye. Optivisor magnification is helpful as is the semi-darkened area. Once visible heat is noticed, things begin to happen quickly. The billet will start to glow brighter, going from a very dull orange to orange to bright orange. It's at this bright orange color that the liquidus state will be reached, and this is evidenced by a wet, shimmery look (Figure 12).

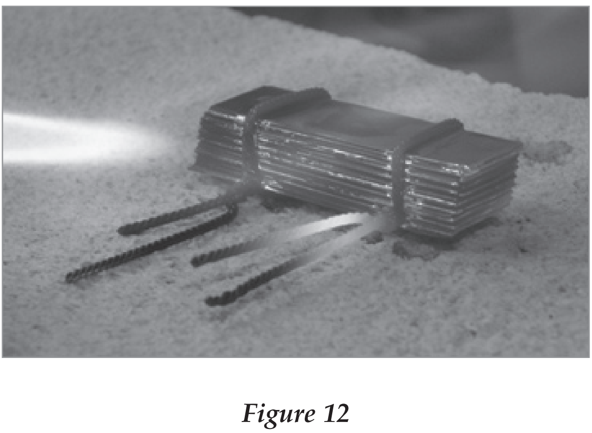

Figure 12

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Magnification is extremely useful and will allow you to notice the very beginning of this liquidus phase. The liquidus phase, once it begins, spreads very rapidly and in a matter of seconds you can be left with a melted pile of metal (worst case), or the layers with the lowest liquidus can melt and flow out from between the layers with the higher liquidus (not the worst case, but pretty bad!!). It is critical to notice the start of the billet bonding and to move the torch rapidly to keep the whole billet at an even temperature once it begins. I heat the long side facing me just enough to notice the shimmer on the surface, then carefully turn the billet 180 degrees using the rotating soldering surface. If turned too quickly, the billet may slide and fan out, ruining the materials. Total torch time is typically under 5-8 seconds per each long side. Then I'll heat the billet from the short end sides for 3-4 seconds per end.

Once this is done, I let the billet cool to the point that the shimmer is gone (this takes from 5-10 seconds), and then flip the billet over and repeat the heating sequence at the lower end of the time scale (5 seconds per long side and 3 seconds per end). The billet is left to cool to black heat on the firing surface and then quenched in water. These times are based on a specific torch type/btu output and billet size, but they give you some guidelines for what to expect while torch firing billets.

Once the billet is quenched, the stainless steel binding wire is removed from the billet. If the billet has been properly fired, you'll have one piece of metal ready to be pickled. After pickling in citric acid solution, the billet is ready for reduction.

Reductions

We chose to reduce the liquid-phase billets using four of the same methods as tested in last year's paper. We wanted an answer regarding the resulting strengths of the materials – would they indeed be stronger than the solid-state diffusion billets as we had suspected and observed in less-controlled manipulations?

In keeping with the low-tech, studio approach to the method, all liquid-phase billets were annealed using flux and the torch with which we fired them. Billets were heated by the same technician in the same darkened area. The flux went through the following stages: first the flux appeared dried, white and crusty; then it looked clear and exhibited surface tension; and finally the flux flowed freely across the surface of the billet. The flow of the flux also coincided with the first appearances of a dull orange color, at which point the torch was removed. When all visible heat was gone, the billets were quenched in water.

Samples of the liquid-phase billets as fired, the solid-state billet as fired, and the billets after reduction were cut for photo micrography by Stewart Grice of Hoover and Strong so we could compare the diffusion zone thicknesses. In addition, the patterned samples were sent to Stewart Grice for photography in order to compare appearances.

The solid-state billet was given an initial cold press reduction from 12mm to 10.6mm (in keeping with the processes detailed in the 2009 paper), then hot forged using a hydraulic press similar to the one commonly used by Damascus steel makers. This is a method used by many mokume smiths, each with their own slight individual variances, and represents the method most typically used in a production environment lacking power hammers or larger hydraulic presses (greater than 50 ton). We do a great deal of hot pressing in my shop and are comfortable with this method. It provides for fast, rapid reduction of billets (typically 30% plus reductions), and we can combine the annealing step with the hot pressing. Billets were coated with a thick layer of boric acid crystals and denatured alcohol and then wrapped flat in stainless steel foil. This package was placed between two 1″ thick A-286 stainless steel plates that had been thoroughly coated with boron nitride and then bolted at all four corners. Little pressure was placed on the billet, the goal being to evenly tighten the plates enough to hold everything together when pressing, not to reduce the billet via cold pressing. The billet was placed into a pre-heated 752°C (1385°F) kiln, and allowed to soak for 60 minutes. The whole assembly was then removed and pressed from 10.6 to 8mm. This was accomplished in the 50-ton press using standoffs and stops matching the height to which the billet was being reduced. After annealing, this process was repeated to reduce the billet from 8mm to 6mm.

Liquid-phase billets were reduced in the following ways from the initial stack height of 10.8mm:

- Hot forging (Billet 1) – This billet received identical treatment to the solid-state billet detailed here previously with the exception of the reductions. The billet began at 10.8mm, was reduced to 8.4 and then to 6mm. In last year's study, this method proved to provide the rapid reduction of a bonded solid-state billet to usable materials as well as produce material of excellent strength necessary to stand up to our bond failure tests.
- Cold forging by hand (Billet 2) – This method is prevalent among beginners as well as commonly used by experts, and figured quite heavily in reducing and testing the bond strengths of liquid-phase bonded billets in our early trials. Hand forging was accomplished using a pair of channel lock pliers (teeth ground off), the anvil, cross peen hammer, blacksmith flatter and sledge hammer. The billet was held in the channel lock pliers and was forged with the cross peen hammer using even, overlapping blows, starting at the outside and working in. When the approximate correct height was reached (within .2 to .3mm), the flatter was held parallel to the surface of the anvil by an assistant and then struck with the 8-pound sledge hammer to even the surface and reach the correct thickness. Following these steps, the billet was reduced to 8.5mm, annealed, reduced to 7.4mm, annealed and finally reduced to 6mm. When used on the 2009 solid-state materials, this method produced billets of excellent strength, although reduction was fairly time-consuming.
- Cold forging using the 50-ton hydraulic press (Billet 3) – When used on solid-state bonded materials, this method produced reduced material of great strength as well. Unfortunately, this process is limited by both the surface area of the billet being processed and the size of the press. The liquid-phase billet was reduced from 10.8 to 8.6mm when we observed that liquid was extruding from between the layers. We removed the billet from the press, examined it, and decided to continue processing. We annealed, pressed the

billet to 7.4mm, annealed, and reduced to 6mm without any further evidence of liquid between the layers.

- Square wire rolling (Billet 4) – We decided to include this method despite the fact that during last year's research the material failed during the early stages of reduction and could not be failure tested with the other materials. My thought was that we could easily see if the liquid-phase bonds were stronger during our early stages of reduction by adding this method. After rolling from 10.8 to 8.5mm, we noticed an apparent delamination at the trailing end but no layer peeling as had been exhibited with the 2009 solid-state material. We decide to continue processing, and annealed and rolled the square wire billet to 7.4mm, then annealed and rolled to 6mm. We continued to see small failures during the course of these subsequent reductions but no layer delaminations as we had had with the solid-state material. The small failures seemed to be caused by the layers curling over each other at leading and trailing edges rather than by any bond issues.

Diffusion Zone Comparisons

Last year's paper dealing with solid-state diffusion bonding showed diffusion zones ranging from 13.1 to 33.98 microns. The measurements of the liquid-phase billets bonded this year were in the range of 10.141 to 31.55 microns. This result was surprising as I expected to see much larger diffusion zones to explain the popular thinking that billets bonded in this method have a 'muddier' look when patterned.

Photo micrography was provided by Stewart Grice of Hoover and Strong. Diffusion zone thickness was measured on un-etched and etched samples. Measurements were taken on as-fired samples as well as samples taken after reduction for both liquid-phase and solid-state diffusion billets. Results are detailed in the chart below (Figure 13). Both liquid phase (lp on the chart) and solid state (ss on the chart) are given as averages between the diffusion zone size as measured etched and un-etched.

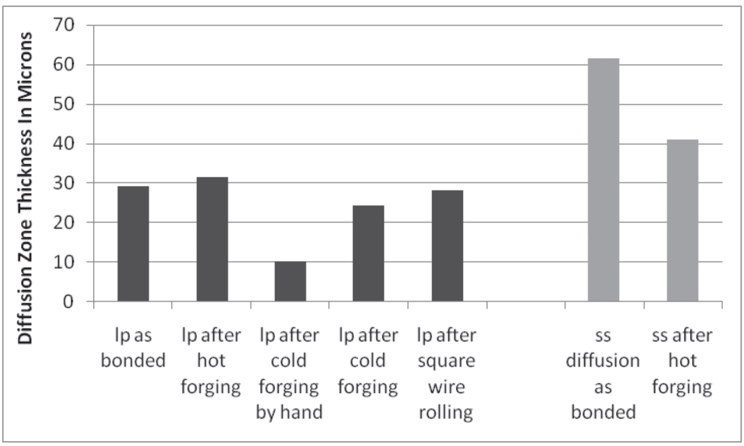

Figure 13

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The hypothesis of much wider diffusion zones in the liquid-phase materials was completely incorrect. In fact, the diffusion zone measurement averages in all cases were smaller than the solid-state diffusion zones. The solid-state zones as measured were larger than those measured for last year's paper. This result is counterintuitive for this material. The only difference in processing from last year came after the initial firing of the billet, and that was the method of annealing.

A Closer Look at the Diffusion Zones

Upon examination, the solid-state diffusion zones seem to be quite even with no porosity visible and have an even color gradient (Figure 14, un-etched; Figure 15, etched). These observations are consistent with what we recorded in last year's paper.

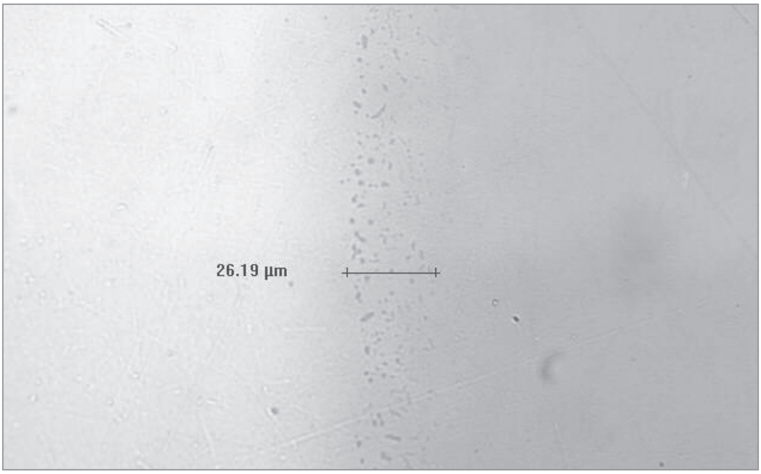

Figure 14 *Solid-state diffusion zone, un-etched*

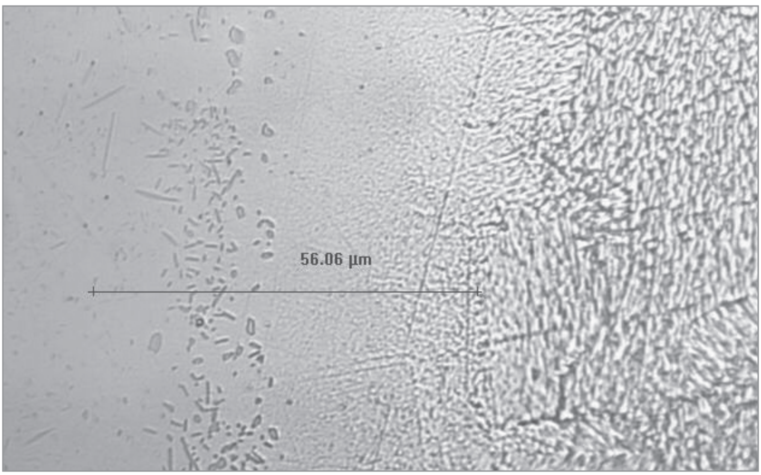

Figure 15 *Solid-state diffusion zone, etched*

Liquid-phase diffusion-bonded billets all had porosity in the diffusion zones. When the silver liquates it expands, and as it solidifies it contracts. This left voids easily seen as dark areas in the photos below. The assumption of makers of mokume gane is that any voids in the bond will cause failures during downstream processing (Figures 16-20).

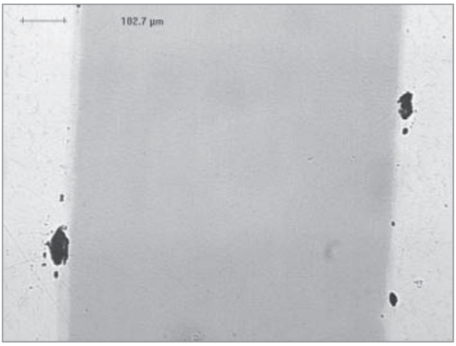

Figure 16 *Liquid-phase billet, as bonded*

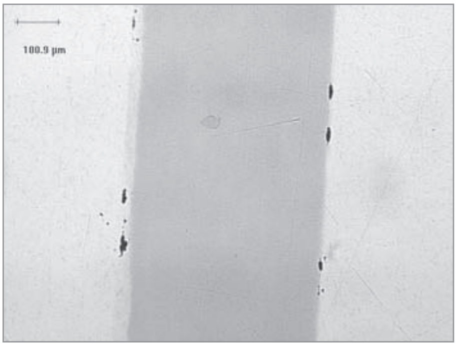

Figure 17 *Liquid-phase billet, hot forged*

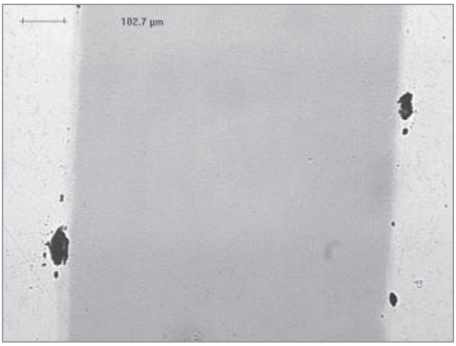

Figure 18 *Liquid-phase billet, cold forged*

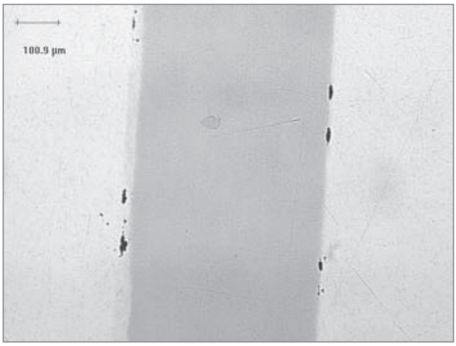

Figure 19 *Liquid-phase billet, cold hand forged*

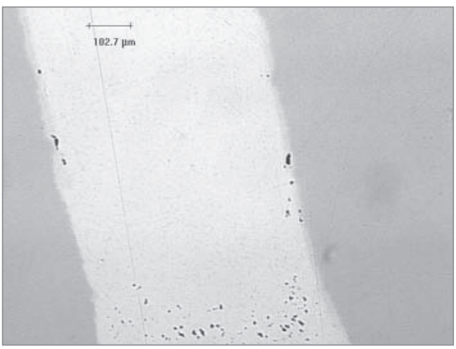

Figure 20 *Liquid-phase billet, square wire rolled*

A very definite medium-gray area in the diffusion zone was also clearly apparent. Perhaps this explains the 'muddy color' noted by previous makers (Figure 21)?

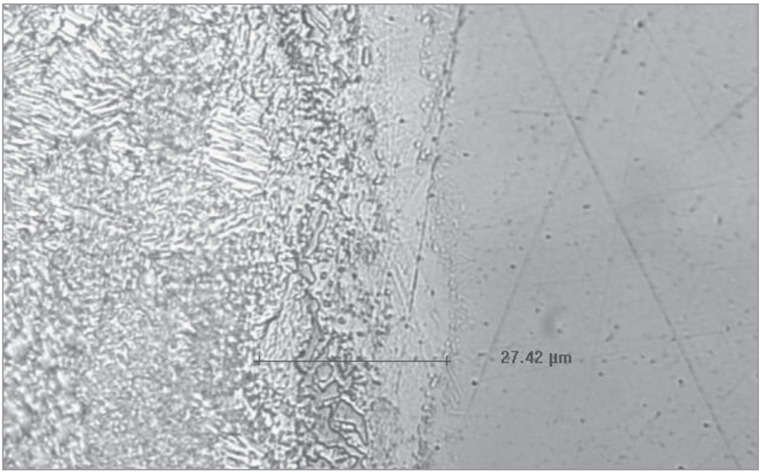

Figure 21 *Medium-gray area in liquid-phase diffusion zone*

As bonded, the liquid-phase billet exhibited an easily measured, clearly defined bond both when un-etched and after etching. After etching it shows a lot of activity in the bond area as well as a darker area β-phase (copper-rich and therefore appears darker than the bulk matrix, Figure 22).

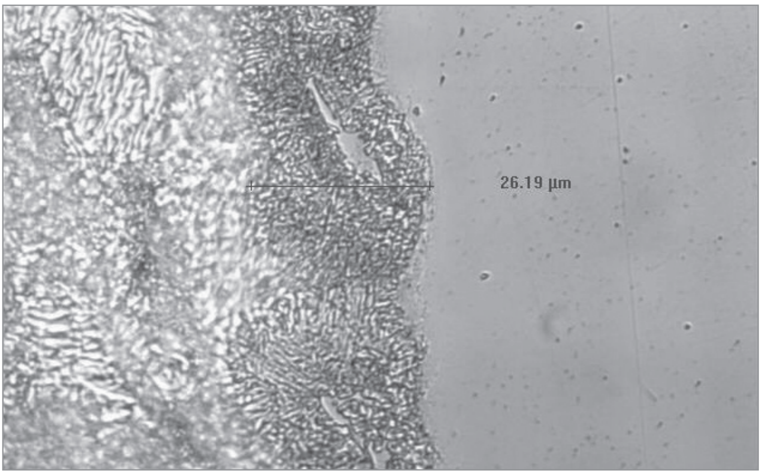

Figure 22 *Clearly defined bond area in liquid-phase billet, etched*

The hot-forged billet exhibited a very uneven diffusion zone. Even after reduction this unevenness still existed, suggesting that if the material is not bonded precisely, then the reduction steps cannot correct this. This finding dovetails nicely with other concurrent research by James Binnion for this year's Santa Fe Symposium, which suggests that reduction is not as important as the initial bond quality of the material. In fact, due to this unevenness of bond zone, when measurements were averaged, it appears to have a bond zone width that is wider than that of the as-fired material even after a 50% reduction. Could this be due to an overheating of the material or an uneven heating of the material while liquid-phase bonding of the billet?

Cold forging by hand reduced the diffusion zone significantly and left a very even-looking bonded area. Although the cold-forged billet had a clean-looking diffusion zone, much tighter than the as-fired sample prior to etching, it seemed to show the same order as other samples after etching as well as the dark line that may contribute to the 'muddy' appearance. (Figure 23 un-etched; Figure 24, after etching).

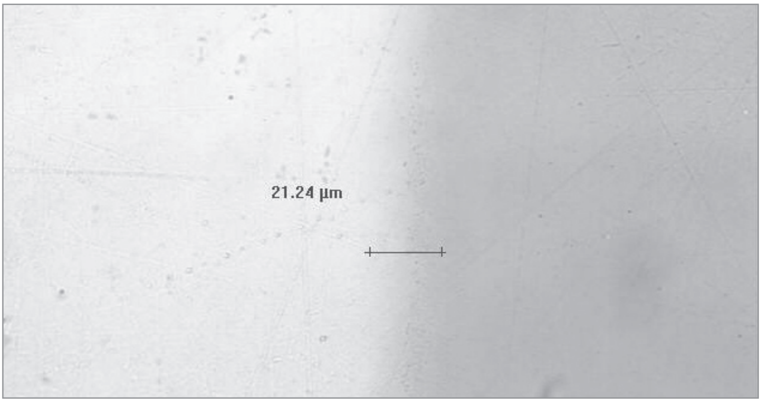

Figure 23 *Diffusion zone of cold-forged billet, un-etched*

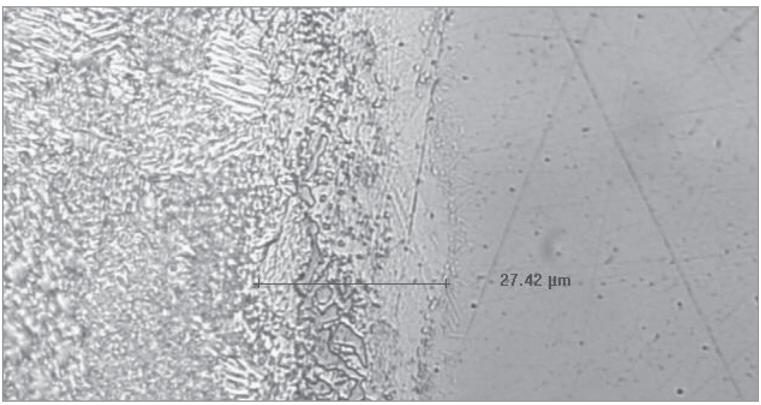

Figure 24 *Diffusion zone of cold-forged billet, etched*

Square wire-rolled materials exhibited an extremely uneven bond zone, similar to the hot-forged materials, with very little reduction in bond-zone thickness (Figure 25, un-etched and Figure 26, etched). Note the dark porosity visible even after reduction. This may again indicate that the downstream processing of mokume gane materials may not enhance the strength of the bond as many makers suppose, and, once again, may indicate the critical factor is the initial bond.

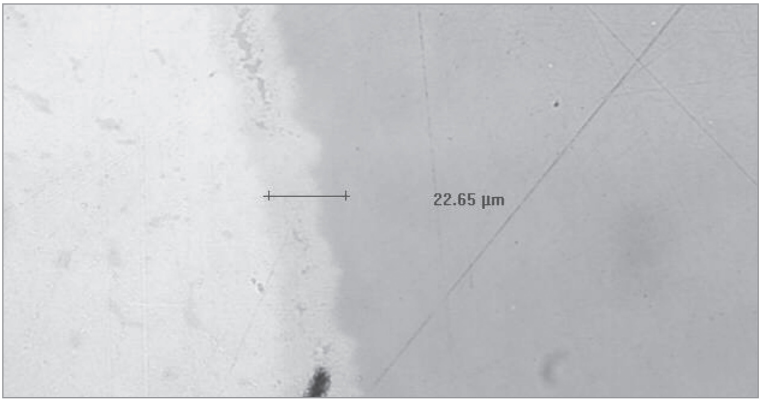

Figure 25 *Diffusion zone of square wire-rolled billet, un-etched*

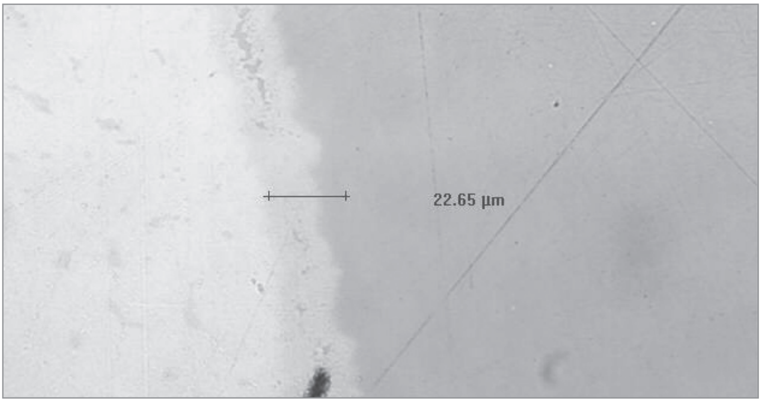

Figure 26 *Diffusion zone of square wire-rolled billet, etched*

Bond Strength Testing

After reduction to 6mm, two samples were cut from each of the four liquid-phase billets that each measured 6 x 6 x 40mm. We used two methods of testing based on the methods devised for the 2009 paper. These methods were developed based on the observations of several makers of mokume gane and the input of a metallurgist.

Ploof

The first method used the rolling mill. Samples were placed with the layers perpendicular to the rolls and rolled with reductions of .2mm taken at each pass. After each pass through the rolls, the samples were examined under 10X for signs of failure. The rolling mill used for these tests is pictured in Figure 27.

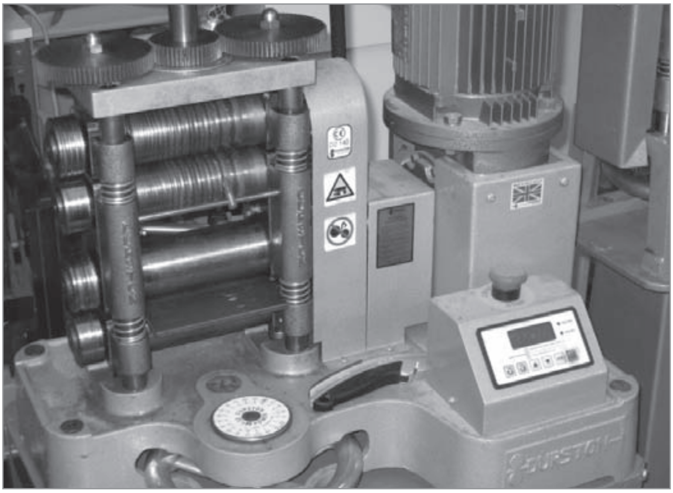

Figure 27 *Rolling mill used to test billets*

The second method of testing used the 50-ton hydraulic press. This test serves to corroborate the findings of the process detailed above and provides valuable information since many makers of mokume gane use the hydraulic press for reductions of stock while patterning to usable materials for fabrication into finished jewelry. Billets were placed with layers perpendicular to the platens of the press, pressed at 5-ton increments and then inspected for failures with 10X magnification after each press. The press use is pictured in Figure 28.

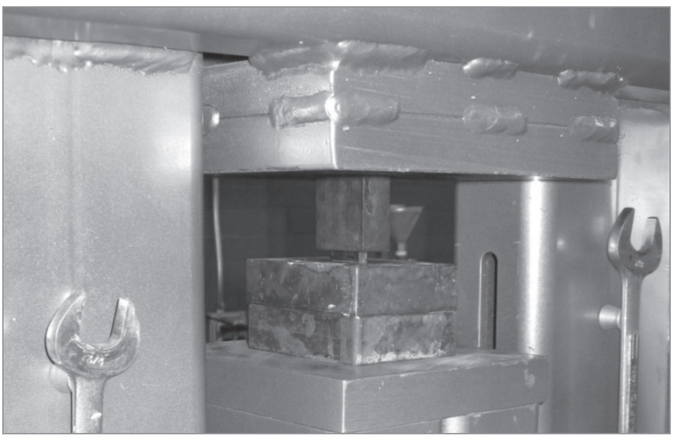

Figure 28 *50-ton hydraulic press used to test billets*

Bond Strength Testing

During testing, a few things happened that were not expected. While rolling to test the bond strength, the cold-forged billet began to delaminate when the thickness of 4.6mm was reached. We trimmed the billet on both ends and examined the delamination. It did appear to have contamination, perhaps from the initial bonding, and we assumed this contamination was linked to the liquid we saw oozing from between the layers during the initial reduction steps as detailed previously. We assume that this liquid was pickle. We took one of the ends and continued processing the material until it finally failed at .4mm in thickness.

The same billet, cold forged, experienced difficulties with the pressing test as well (not unexpected after the rolling results). Failure occurred after 5 tons of pressure at the center of the billet. Once again, the section was trimmed out and the remaining end was processed to 30 tons of pressure.

The following graphs show the results of the failure testing of the liquid-phase billets compared to the solid-state billets from the 2009 paper. Figure 29 (Billet failures, rolled) shows the results of the samples of each billet that was rolled to failure. Simply put, the lower the bar on the chart, the longer the material lasted during testing.

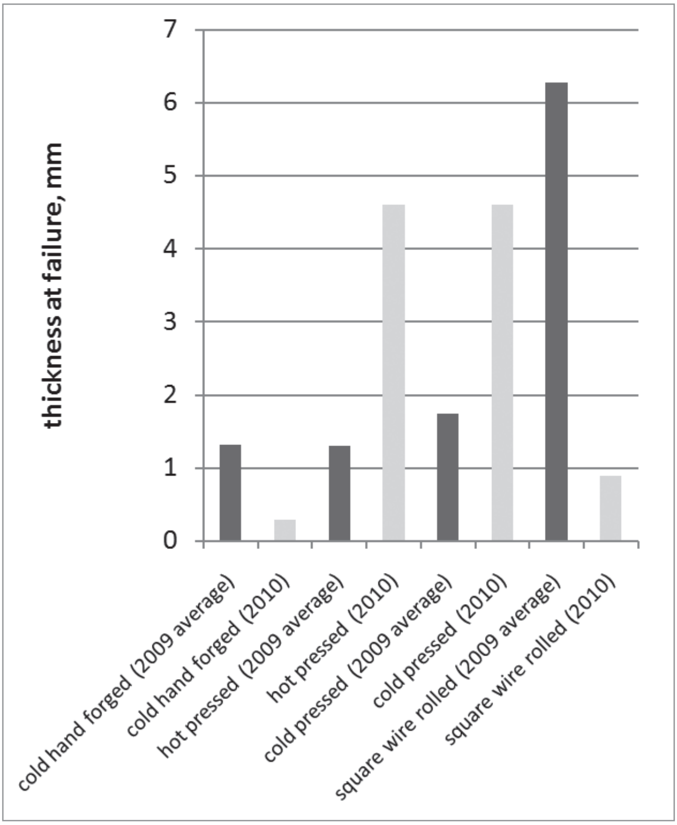

Figure 29 *Billet failures, rolled*

Figure 30 (Billet failures, pressed) shows the results of the cold-pressed to failure samples. The results are charted by the amount of pressure in tons (gauge reading, not actual surface pressure on the samples). The higher the bar, the stronger the material.

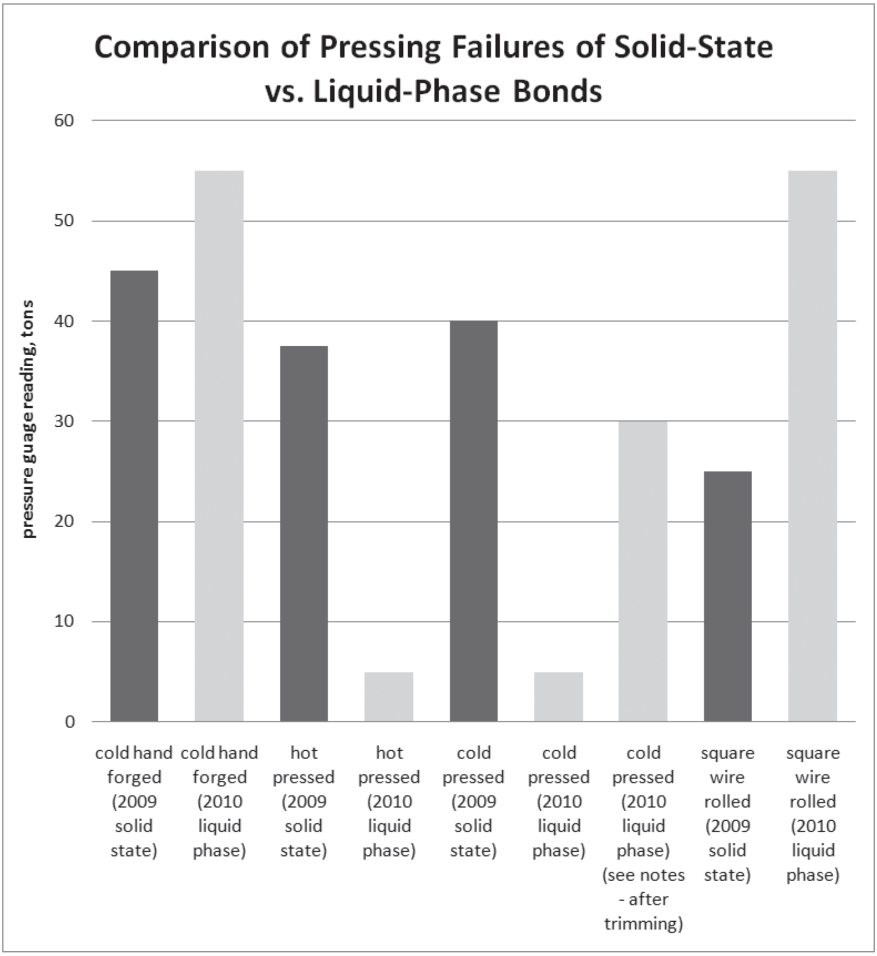

Figure 30 *Billet failures, pressed*

Several results were realized during the testing. Repeatability in torch firing can be difficult. This method does work but because so much is operator and environment dependent, it can be quite difficult to get exact results. Of the six 14K palladium white gold and silver billets, two exhibited failures during processing. The cold-forged material used for bond testing had a delamination caused by what I suspect to be under firing of the billet, and the billet used to make patterned sheet had a failure that became apparent well into the patterning stages (Figure 31).

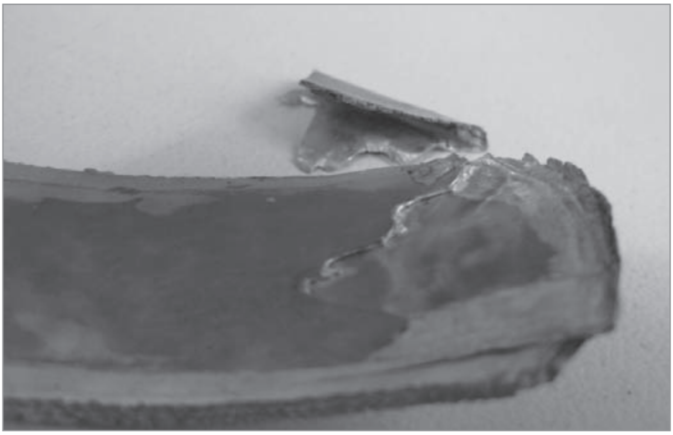

Figure 31 *Patterned sheet failure*

But some of the most interesting results were in the way of billet strengths. My predictions were that all of the liquid-phase billets would exhibit strength greater than the solid-state billets tested last year. This was based on my personal experience when making liquid-phase billets, as well as the experience of other makers of mokume gane. And it was incorrect.

Solid-state diffusion-bonded billets from 2009 exhibited their best strengths when cold hand forged, exhibited great strength when hot or cold forged using the hydraulic press, and had very poor strength when rolled from initial size to square wire. Failures occurred prior to reducing the material far enough for failure testing with one group of samples, and the second group fared very poorly, with minimal reduction possible prior to failure. The liquid-phase billets from this year's study exhibited their best strength when reduced using cold hand forging and square wire rolling – a result completely contrary to what one could logically expect in the case of square wire rolling.

Both hot and cold forging of the liquid-phase billets using the hydraulic press yielded material that, when rolled, failed much sooner than its counterpart solid-state diffusion material from last year. Part of this may be due to two observations made previously—the hot-forged materials had a very uneven diffusion zone layer, and the cold-forged materials had an early failure that seemed to be caused by contamination of the sheets. Once the failure was trimmed, however, the cold-forged material was processed and fell in between hot-forged and the two methods that produced the strongest material, cold hand forging and square wire rolling. But I don't think the early failure can be blamed entirely on the uneven diffusion zone, as the square wire-rolled material exhibited great strength and also showed uneven diffusion zones (see Figures 29 and 30).

Addressing the issue of appearances, to the casual observer as well as to the trained eye, the layers do not look muddy. In fact, as observed, the layers of the solid-state material appear to be less crisp. Figure 32 shows a 50X view of solid-state bonded patterned material, and Figure 33 shows the liquid-phase patterned material. Both samples were prepared up to 1200 grit paper and then polished with white diamond followed by blue rouge prior to examination by naked eye and under the USB microscope at 50X.

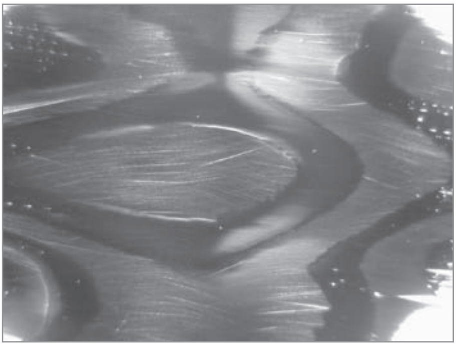

Figure 32 *Solid-state-bonded patterned material, 50X*

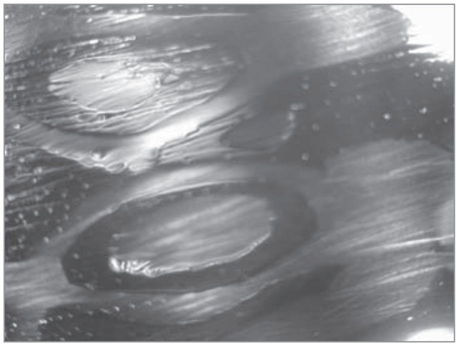

Figure 33 *Liquid-phase patterned material, 50X*

Figure 34 shows another area of the solid-state-bonded patterned material sample at 200X, while Figure 35 shows the liquid-phase material taken with our in-shop USB camera. In both comparisons, the layers of the liquid-phase materials appear to be more crisp and not muddy.

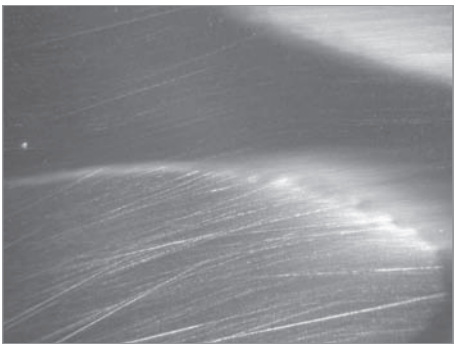

Figure 34 *Solid-state-bonded patterned material, 200X*

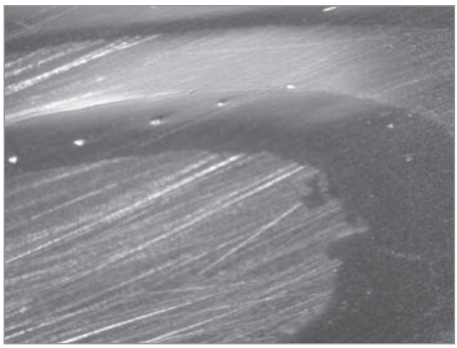

Figure 35 *Liquid-phase patterned material*

Figures 36 and 37 are taken by Stewart Grice of Hoover and Strong. Figure 36 is the solid-state material, while Figure 37 is the liquid-phase material. Both are at 50X and again, the solid-state diffusion zone looks softer and more muddy than the liquid-phase diffusion zone.

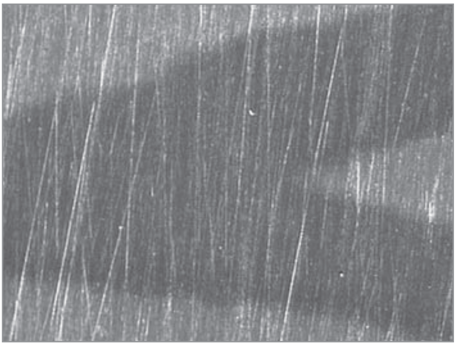

Figure 36 *Solid-state material, 50X*

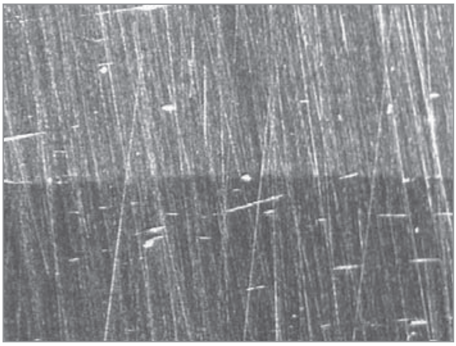

Figure 37 *Liquid-phase material, 50X*

Conclusions and Recommendations

1. Liquid-phase billets as bonded in our method are fast, with firing times in minutes rather than hours, and are easy as they require little in the way of special tooling to make.
2. Repeatability can be difficult as exhibited by two failures in our group of six billets.
3. Control of both temperature and time remains important. Perhaps with a better method of temperature and time control and a greater understanding of how the variables can affect the resulting billets, repeatability could be improved, leading to less material failure.
4. The weakest materials were those reduced by hydraulic forging, whether cold or hot. Both materials failed at nearly the same instance when rolled with layers perpendicular to the rolling mill rollers, and both delaminated at the same pressure when press tested. Although, after trimming away the delamination of the cold-forged billet, which seemed to have been caused by contamination in the early stages of bonding, the material did exhibit trength a bit greater than that of its hot-forged counterpart.
5. The strongest material was produced by cold hand forging, and this result is identical to last year's testing of solid-state diffusion-bonded materials. Material of nearly the same strength was produced by square wire rolling, a method of reduction that, when used last year to reduce solid-state diffusion-bonded materials, failed either prior to testing or very early in testing. This was completely unexpected.
6. For the modern manufacturer of mokume gane, the best repeatability seems to come from solid-state diffusion billets. This method also lends itself to larger billets in a smaller shop.
7. Cleanliness, although assumed to be less important for liquid-phase billets, may be equally as important for firing quality materials regardless of bond type.

8. Billet size is restricted when torch firing at the bench. Larger billets could potentially be fired if a torch head could be adapted, but previous results show that as billets get larger (in both surface area and thickness), one of two things typically happens. Either they appear to bond well before further processing shows that the center of the billet did not bond, or more heat is used, melting outside edges and still resulting in a failure to bond in the center.
9. Initial bond strength for liquid-phase billets may be more and less important than for solid-state diffusion billets. Note the earlier observations of porosity, which in solid-state billets is believed to lead to material failures, which did not seem to affect bond strengths in our testing. But we did also note very uneven diffusion zones, which may have played a part in the failure of the hot-forged billet but which did not seem to affect the square wire-rolled billet.

For larger production, I consider the solid diffusion-bond method to be superior due to repeatability as well as the ease of scaling in the well-equipped shop. However, if the repeatability issues could be addressed, the liquid-phase method may well produce stock that can be quickly and rapidly reduced to usable materials for small jewelry objects. Two rings can easily be made from a billet the size that we bonded for these tests as well as patterned sheet in dimensions of ~102mm x 25mm x 1mm (~4″ x 1″ x 0.040″). Given the rising cost of precious metals, this approach has real merits in terms of limiting the amount of precious metal necessary to have on hand in the currently volatile market.

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2. Chris Ploof, "Mokume Gane Billet Reductions and Their Effects on Bond Strength," *The Santa Fe Symposium on Jewelry Manufacturing Technology 2009*, ed. Eddie Bell (Albuquerque: Met-Chem Research, 2009).

3. Steve Midgett, *Mokume Gane, A Comprehensive Study*, 1st ed. (North Carolina: Earthshine Press, 2000).