VACUUM PROCESS ENGINEERING

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ACTIVE METAL BRAZING JOINT STRENGTH CORRELATION

•• Post Braze Zirconia Tint as a Qualitative Indicator of Braze Joint Strength ••

ABSTRACT

Brazing of stainless steel to zirconia with a titanium-bearing active alloy results in a visible darkening, or tinting, of the zirconia member. It was found that the degree of tinting has the capacity to be used as a reliable proxy for strength of the braze joint. A set of 16 brazed coupons were measured for post braze tint and braze joint strength. The results showed a direct statistical correlation with an R^2 value of 0.74. Based on this data, optical measurement of the post-braze zirconia surface darkness has the potential to be used as a quality assurance tool for improving braze yield in large production batches.

ABOUT THE PROCESS

Active metal brazing is a material joining process worth considering when at least one of the joint materials of interest is a ceramic. While typical brazing processes are only effective when the joint surfaces are metallic, active metal brazing allows for brazing directly to a wide variety of oxide, nitride, and carbide compounds usually in a single process step (1).

The process of active brazing is similar to a standard vacuum or inert atmosphere brazing process and is thus readily adaptable to standard vacuum brazing equipment. One key difference is that active braze filler metals (BFM) necessarily include an "active" element, e.g., titanium (and often traces of "support" elements), which are usually alloyed into the filler metal. The active element works by diffusing to and reacting with the ceramic surface during brazing. A well-designed braze process will result in the formation of a continuous transitional layer – the so-called reaction layer – that provides a wettable surface of semi-metallic character (2). The formation of this reaction layer then allows for the molten BFM bulk, usually of fairly typical braze alloy composition, to form a coherent braze joint between the member components.

Figure 1: Square of active braze foil fused onto alumina at approximately 940ºC in partial pressure inert atmosphere

INTRODUCTION TO THE CASE STUDY

The application of active metal brazing is not without its particular challenges. Achieving good results can be difficult since the process tends to be more sensitive to parametric variation than more common types of brazing. For instance, the formation of a sound brazement is contingent upon the formation of a thin reaction layer from a relatively small percent of alloyed active element (usually <3%). The thickness of a reaction layer that will provide good joint strength is usually within a small window – typically no more than a few microns thick (2). As a result, a relatively small perturbation in the amount of available active alloy at the ceramic surface can hinder the quality of the braze joint by altering the character of the reaction layer.

Given the greater process sensitivity, one of the key challenges with active metal brazing is producing high yields and consistent joint quality. An active brazed component with a poor quality reaction layer may not be easily distinguishable from a part with a well-formed reaction layer. Both good and bad parts may appear fully brazed with visual indication of alloy filleting. To determine the joint strength, parts usually must be sacrificed by destructive testing. Since parts for service/delivery to customer cannot be destructively tested, witness coupons or statistical batch testing are required for quality assurance in production. However, due to the sensitivity of active brazing, parts from the same batch may not be of homogenous quality with the potential result being that poor quality parts can pass undetected through inspections. This case study will show that post-braze zirconia tint is likely a good auxiliary measure for joint strength that can indicate strength directly without destructive testing.

DESCRIPTION OF THE EXPERIMENTS

The experiment described in this case study was a development effort to join a zirconia ceramic component to stainless steel by active metal brazing. WESGO Silver-ABA^{TM} foil preforms of 0.002" and 0.0028" thick were pre-placed between the components and then pressed together by a nominal weight. The braze fixturing consisted of a stainless steel base plate with tungsten alignment pins and a hole into the center of mass to accept a 1/16" type K thermocouple.

16 braze coupons were created using multiple varied parameters such as ramp rate, soak temperature, soak time, deadweight, alloy lot/thickness, and atmosphere type. The parameter set for each run was designed to test different brazing conditions and their effect on the braze strength. All braze runs were done in all-metal cryo-pumped vacuum furnaces using PID controllers, 4 zone thermocouples, and 1 or 2 work thermocouples.

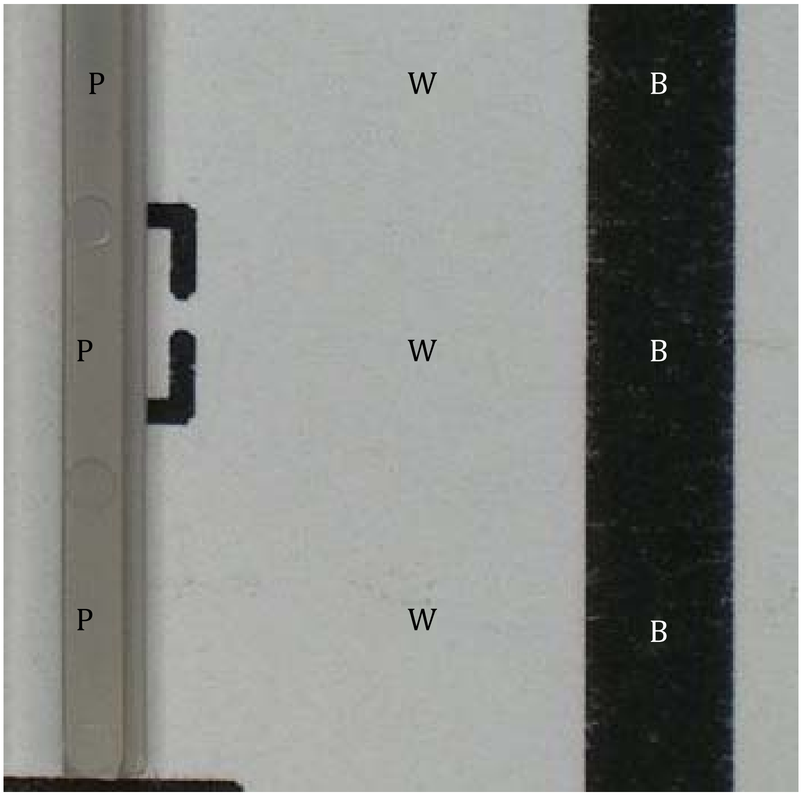

Figure 2: Measurement key for zirconia substrates. Part is at the left. Black line and white paper on which it is printed are both used for calibration. Letters denote where measurements were taken.

After brazing, the parts were first measured for the degree of tint. This was done using a standardized digital image of the zirconia component by way of a graphics program to extract color-space information. The specific measurement taken was the "Value" component of the

HSV (Hue, Saturation, Value) color-space. This measurement results in 0% for full black and 100% for full white, effectively a scale of dark to bright. To accommodate for variations across the digital images and from image to image, the brightness was sampled at several places on the zirconia surface and on several black and white calibration regions (Figure 2). The color value was then linearly interpolated using a scale of 0-100 based on the calibrated offsets.

For measuring the strength, a scale of 1 to 5 was defined for assessing the peel test strength. The peel test was done either by hand or using a small pair of needle-nose pliers. The qualitative scale is as follows:

1. No braze or very easily unzips
2. Low resistance. Can be peeled entirely by hand
3. Medium resistance. Can be peeled mostly by hand
4. High resistance but with discontinuous fracture in the BFM. Pliers mostly required
5. High resistance with uniform fracture in the BFM. Pliers required throughout.

RESULTS

The tint and strength results varied substantially from run to run. It is outside the scope of this paper to analyze the reasons for the wide range in results, but suffice it to say that the variation appeared to have been caused by several process parameters.

All zirconia substrates in contact with active braze alloy were darkened during the braze cycle. The darkness change can be observed throughout the entire zirconia component and appears to be fairly constant regardless of the proximity of the braze joint. A cross-section of the part showed that the color change also occurs in the interior of the material.

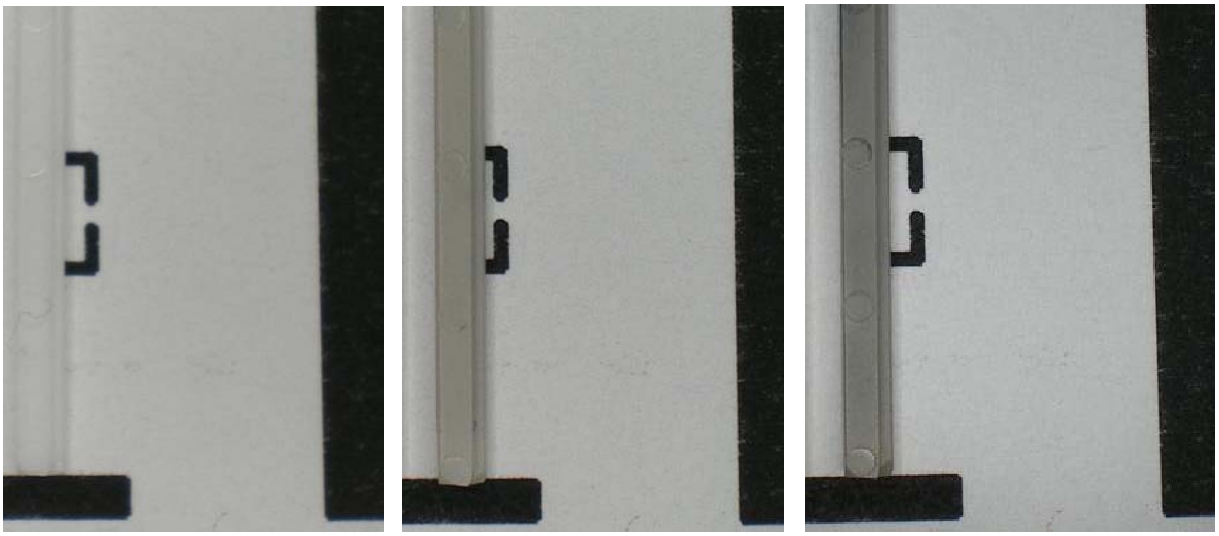

Figure 3: Three zirconia ceramics of different tints. The ceramic at the far left is pre-braze.

A comparison of the HSV brightness and the qualitative strength values reveals an inverse relationship between the HSV-value of the post braze zirconia components and the joint strength of the brazement. In general, the lower the HSV-value (or darker the tint) of the post-braze zirconia component, the stronger the joint strength. A small amount of data scatter is evident; however there are no large divergences, and a statistical R^2 value of 0.74 shows strong determination of the data set.

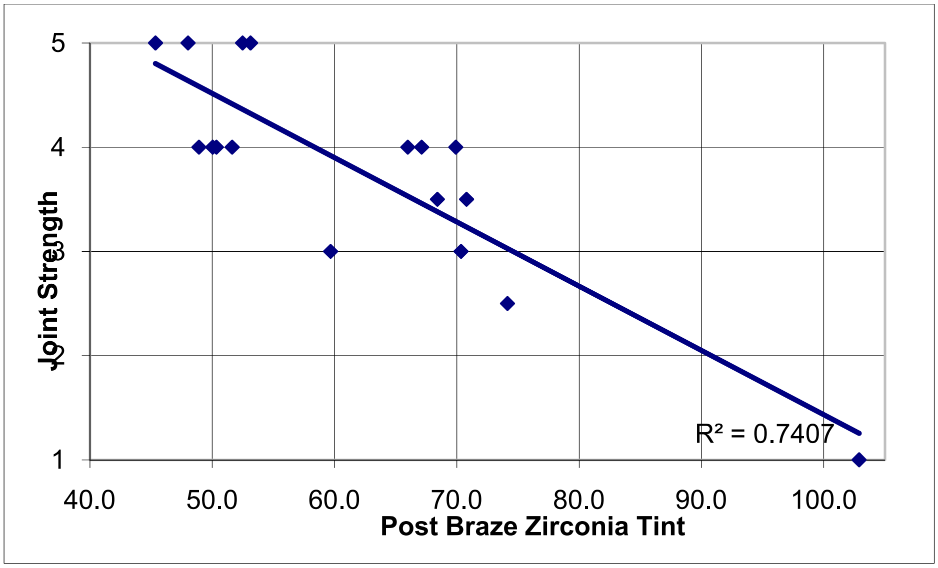

Figure 4: Graph of the Joint strength versus the zirconia brightness value shows a clear correlation between darker "tint" and joint strength. The data point at the far lower right was exposed to a brazing cycle only. The reason this data point is greater than 100 is because it measured a higher brightness value than did the white (calibration) paper.

Figures 5: Parts are arranged by darkest to lightest (zirconia tint) from top to bottom. The middle part is shown midway through the peeling process and BFM can be observed on both the zirconia and the stainless surfaces such that the weak point is the alloy itself (as desired). The darker residue in the bottom part (with the lighter zirconia) shows only a small amount of BFM residue. The darker residue may be the result of an oxide and/or Ti intermetallic formation caused by poor contact of the BFM preform with the zirconia substrate.

Fracture surfaces were inspected after peel testing to confirm the qualitative strength results. Strong braze joints resulted in fracture of the BFM during peeling, while a weaker joints resulted in fracture at the ceramic-to-BFM interface. The results of the qualitative strength measurements also correlate to the amount of fracture occurring within the BFM.

DISCUSSION

Although a strong correlation was reported between post-braze zirconia tint and joint strength, the strength of the data might be questioned based on several factors. The peel strength of the joint was done manually and sometimes several days apart. To counter any inconsistencies this may have introduced, the resolution of the scale was made deliberately coarse and also backed by visual standards. This made it difficult to misinterpret the strength of a brazed joint. Secondly, the digital imaging system used to record the images for darkness measurements was used in a room with exterior windows and potential sources of “light noise”. This also was countered by using a set light source and also black and white standards to calibrate the measurements.

On the other hand, the correlation was supported by a good amount of data and a relatively high coefficient of determination value, R^2. Also, because a number of different process parameters were varied (ramp rate, soak temperature, soak time, deadweight, alloy lot/thickness, and atmosphere type), the correlation is seen to be applicable for a wide variety of process parameters that may influence joint strength. This lends to the idea that the darkening is directly related to the characteristics that give strength to the joint. Another factor used to support the results was the visual observation of the fracture joints. Qualitative evaluation of the brazed joints showed a greater amount of residual braze alloy on the zirconia member for stronger assessed joints, and vice versa.

The rigor of the experiment was sufficient to produce meaningful results, however these results do not imply that there is no case in which a post-braze zirconia joint with dark tint could in some way be of poor quality/strength. Active braze joints will exhibit poor strength when the reaction layer grows to an excessive thickness. As a result, a joint supplied with too much titanium due to excessive time at temperature can easily result in a dark zirconia tint and yet show a poor quality joint. While it was not observed in this experiment, this case might in fact produce a tint above some threshold value upon which a reduction in joint strength can also be correlated with.

The theory behind the post-braze change in darkness of the zirconia components is reportedly caused by a slight change in the stochiometry of the zirconia when reacted with titanium (3). The change in darkness, therefore, indicates that titanium is diffusing into and reacting with the ceramic substrate – the desired condition. This fact seems to explain the correlation observed in the data between strength and darkness, however, it could also be argued that a bulk darkening of the zirconia member, as observed, is the result of a titanium depletion at the joint interface caused by the titanium diffusion into the zirconia substrate. The preference is clearly to have titanium active at the interface, and in this case any additional diffusion of titanium into the zirconia bulk may simply have to be overcome by a greater amount of Ti in the braze filler alloy. This is supported by literature that shows greater joint strengths when using alloys having higher Ti composition than typically used for other common oxide ceramics.[3]

CONCLUSION

When brazing Zirconia using Ti-bearing active alloys, a visual darkening (or a reduction in color-space value) of the zirconia component occurs when the Ti active element reacts with the zirconia. In the experiment discussed herein, the amount of darkening in the zirconia was shown to correlate well with the strength of the braze joint. This can be a very useful visual indicator of joint strength and has potential as a quality assessment tool. This measurement tool could allow a quality inspector to reject poor quality brazed parts (parts with zirconia tint that are too light) that would otherwise have slipped through inspections. A more precise qualitative measurement could even be sufficient as a basis for a 100% non-destructive inspection plan.

Darkness measurements can also be a very useful troubleshooting or development tool. Comparing post-braze zirconia darkness values in test or qualification runs can give good indication of titanium activity at the joint interface, a critical component of active brazing kinetics.

1. H Mizuhara, E Hubel, T Oyama, "High-Reliability Joining of Ceramic to Metal," Wesgo Division, GTE Product Division, Belmont, CA 94002

2. David M. Jacobson, Giles Humpston, "Principles of Brazing," ASM International, United States of America (2005)

3. W.B. Hanson, K.I. Ironside, J.A. Fernie, "Active Brazing of Zirconia," Acta Materialia 48 (2000) 4673-4676