BY ALEXANDER E. SHAPIRO

Q: What etchants do you use to etch brazed joints to visualize the joint metal for metallography using an optical microscope? For instance, stainless steel brazed with BAg-24, or titanium Grade 5 brazed with TiBraze200? I cannot find etchant formulations in the literature, and I am having a hard time seeing the brazed joint properly. Many times we don't have time to send samples for electron scanning microscope study. I may be overetching my samples, but I want to make sure that I am seeing the correct things.

A: You are right. We rarely see optical metallography of brazed joints, etching procedure, and etchant formulations in the literature of the last two decades. Electron scanning microscopy is mostly used today, which does not require etching. However, for practical purposes, many companies still use optical metallography of polished and etched brazed joints. The following offers some practical information about etchants and other recommendations related to the polishing and etching procedure of cross sections of brazed joints.

Etchant compositions for visualizing the microstructure of the most popular groups of brazing filler metals are presented in Table 1. Some etchant compositions and etching times are given in wide ranges. This is because successful etching depends on many factors, and the process parameters should be adjusted experimentally. This is not difficult to do, usually two or three iterations are sufficient.

The most important factors that affect the etching process and reveal microstructure of both the joint metal and the base metal interface are the quality of polishing and the composition of the etched alloys. The base metal and the joint metal often have different hardnesses; therefore, polishing should be done without a relief (or "step") from the base metal surface to the joint metal surface. In order to avoid this step, we recommend using only fine abrasive papers, changing the direction of polishing by 90 deg every two to three minutes, and frequently rinsing the polished surface under a tap-water stream to remove abrasive grains that can get stuck in soft joint metal. Final polishing with a diamond paste is used in cases where there is a big difference in hardness of the base and joint metals.

An example of the microstructure of a titanium joint brazed with AWS BTi-5 (Ti-20Zr-20Cu-20Ni [wt-%]) is shown in Fig. 1. The etching was done using Reagent #5. The etchant revealed the grains of the

Table 1 — Etching Procedure and Reagents for Revealing Microstructure of Brazed or Soldered Joints of Most-Used Base Metals and Filler Metals

No.	Base Metal	Brazing Filler Metal	Etchant Formulation	Etching Procedure
1			$(NH_4)_2S_2O_8$ 1–20 g, H_2O up to 100 mL. Add 3–5 drops of a saturated ammonia solution immediately before application.	Immerse for 5–10 s. Rinse in cold water.
2	Stainless or carbon steels Cu alloys	Ag-based brazing filler metals	$FeCI_3$ 2–5 g, H_2O, 100 mL (for joint metal), ethanol 100 mL (for steel). Add 20–30 mL H_2O_2 immediately before etching joints of copper alloys.	Deposit etchant for 10–60 s. Rinse in cold water.
3			Acetic acid 5–10 mL, ethanol up to 100 mL	Rub with a wad or Q-tip® for 20–120 s. Rinse in cold water.
4	Stainless or carbon steels Cu alloys	Cu-based brazing filler metals; Sn-Pb solders	Step 1: 4% solution of HNO_3 in ethanol Step 2: 50–60% solution of ammonia in water	Immerse for 5–10 s. Rinse in cold water.
5			HF 2–5 mL, H_2O 100 mL (If a dark shadow appears, immerse in 5% HNO_3 solution for 2–3 s.)	Immerse or rub with a wad or Q-tip® for 10–30 s. Rinse in warm water.
6	Ti alloys	Ti-based brazing filler metals	HF 1–5 mL, HNO_3 1–5 mL, H_2O 89–90 mL. Use hot (60°–70°C) if cold etchant is not effective.	Immerse or rub with a wad or Q-tip® for 2–3 min. Rinse in warm water.
7			Kroll's reagent: HF 4 mL, HNO_3 22 mL, H_2O 100 mL	Immerse or rub with a wad or Q-tip® for 2–10 s. Rinse in warm water.
8	Al alloys	Al-Si brazing filler metals	HF 1 vol part, HNO_3 2 vol parts	Immerse or rub with a wad or Q-tip® for 5–10 s. Rinse in warm water. Dry with ethanol.
9	Cu or Steel	Sn-Pb, Sn-Ag, and Sn-Zn solders	HNO_3 2–6 mL, ethanol 94–98 mL	Immerse or rub with a wad or Q-tip® for 20–60 s. Rinse in cold water.

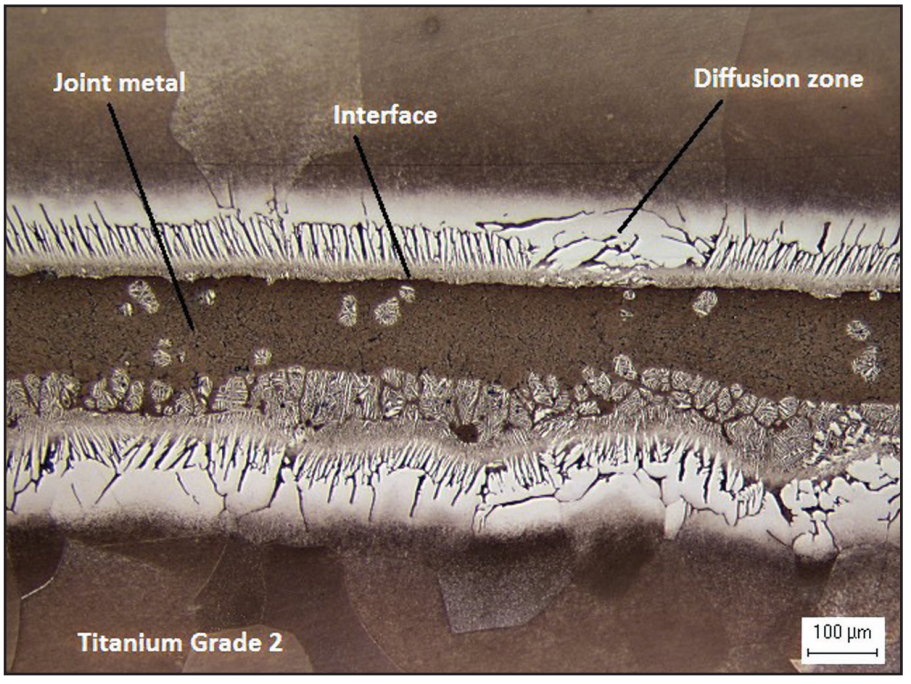

Fig. 1 — Microstructure of titanium joint brazed with BTi-5 filler metal. Etched 30 s with HF 2–5 mL, H_2O 100 mL (Etchant #5 in Table 1).

base metal and width of the diffusion zone (white area), the formation of a typical needle-like structure of Ti-Zr-Cu solid solutions, a thin Ti_2Cu intermetallic layer along the interface, and grains of TiZrNi phase in the joint metal matrix. The structure of the joint metal matrix is not as clear as other characteristics, but at least we can be sure that there are no defects such as pores or cracks.♦

References

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2. Baeslack, W. A., McQuay, P. A., Lee, D. S., and Fletcher, E. D. 1993. Metallography of gamma titanium aluminides. *Materials Characterization* (31): 4, 197–207.

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