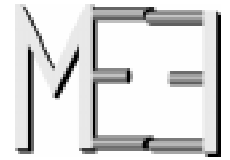


# LABORATORY REPORT



Materials Evaluation and Engineering, Inc

Dr. Otto Focus  
Research Scientist  
Corporation ABC  
123 Industry Road  
Springfield, MN 12345

Report Date: April 22, 1998  
Date Submitted: April 20, 1998  
MEE Project: ZZ0001  
Sample ID: Ticusil braze joint  
P.O. No.: 12345

**PROJECT TITLE:** Evaluation of Braze Joint

## INTRODUCTION

A sample of tungsten metal brazed to ceramic substrate was submitted for evaluation of the braze joint. A characterization of the braze phases and interfaces with the tungsten and ceramic was requested. The braze material was identified as Ticusil braze alloy (68.8% silver, 26.7% copper, 4.5% titanium). The sample was identified as part number AB123.

## SUMMARY AND CONCLUSIONS

The braze microstructure consisted of a primary silver-rich phase and a secondary copper-rich phase. Blocky, dark islands were observed throughout the braze section. Each of these islands had a dark core with many cracks. This core was composed primarily of titanium and nitrogen, possibly in the form of titanium nitride. The brittle nature of the titanium nitride intermetallic would also be consistent with the cracked appearance of the core.

Circumscribing the core for each island were four layers. Moving out from the core, each layer appeared slightly brighter in the backscattered SEM image than the previous layer. These layers were composed of titanium and copper with a small concentration of silver. Each layer had progressively higher concentrations of copper and silver, and less titanium and nitrogen than the previous layer.

A titanium-rich layer was observed at the braze/ceramic interface. No such layer was observed for the braze/tungsten interface.

## TEST PROCEDURES

The sample was mounted in a castable epoxy mounting material to provide across section through the braze joint. The mounted sample was metallographically ground and polished to a plane near the center of the braze joint. The prepared sample was carbon coated prior to analysis to provide a conductive film to allow examination with the scanning electron microscope (SEM) in high vacuum mode, which maximizes image and x-ray resolution.

The braze joint cross section was examined by SEM, and electron micrographs of typical areas were acquired. The backscattered electron imaging (BEI) mode of the electron microscope was used for the examination. In BEI mode, the brightness of particular sample features corresponds to the mean atomic number of the elements composing those features. Thus, features composed of heavier elements appear brighter than features composed of lighter elements.

Qualitative and semi-quantitative chemical analyses were performed on the braze phases using energy dispersive x-ray spectroscopy (EDS). EDS X-ray mapping was also performed on a selected typical area of the braze joint. With x-ray mapping, the brightness of image features corresponds to the relative concentration of the element mapped in that particular image. Thus, brighter areas of the x-ray map image represent a higher concentrations of the element being mapped.

## RESULTS

Electron micrographs of typical areas of the braze joint are displayed in Figures 1 and 2. The braze consisted primarily of one bright phase (henceforth labeled phase A) with several blocky, darker islands distributed through it. At the top and bottom end of the joint, the braze had a more eutectic morphology, with phase A surrounding islands of a darker phase (henceforth labeled phase B). The braze appeared to have flowed into and wetted even the roughest areas of the ceramic substrate. No voids were observed in the braze.

EDS analysis of phase A detected primarily silver, with lower concentrations of carbon, oxygen, and copper, Figure 3. Analysis of phase B detected primarily copper, with lower concentrations of carbon, silver, and titanium, Figure 4. These differences in the silver and copper concentrations between the two phases would account for the brightness difference observed in the BSE images. The carbon detected in these and subsequent analyses is probably due mostly to the carbon film deposited on the sample for conductivity.

The blocky, darker islands distributed throughout the braze appeared to be composed of four layers around a core material, Figure 2. The core material was the darkest and had several cracks. Each subsequent layer going out from the core was slightly brighter than the previous layer. The layers were labeled 1 through 4, with layer 1 being adjacent to the core, and layer 4 being farthest from the core.

EDS spectra for analyses of the core material and the four layers are displayed in Figures 5 - 9. Carbon, nitrogen, copper, silver, and titanium were detected for each analysis. The core material was primarily titanium and nitrogen, with very little copper and silver. Analysis of the four layers going outward from the core detected progressively higher concentrations of copper and silver, and less titanium and nitrogen than the core material.

Semi-quantitative results for the braze phases, core material, and layers 1 through 4 are displayed in the following table. Because EDS has poor sensitivity to light elements, accurate quantitative

results for nitrogen were not possible. Therefore, the table shows only the relative atomic concentrations of titanium, copper, and silver.

Element	Composition, relative at%						
	Matrix Phase A	Matrix Phase B	Island Core	Island Layer 1	Island Layer 2	Island Layer 3	Island Layer 4
Titanium	<0.1	0.8	98.0	77.0	51.7	44.1	23.0
Copper	8.0	90.9	1.2	21.3	45.6	54.4	74.0
Silver	92.1	8.3	0.8	1.7	2.7	1.5	3.0

A dark layer was observed along the braze interface with the ceramic, Figure 10(a). Analysis of this layer detected significantly more titanium than for either of the braze phases, Figure 11. This indicated that the layer was probably titanium from the braze which had segregated to the interface.

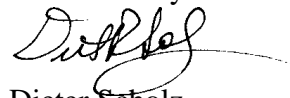
No unique layer was observed at the braze/tungsten interface, Figure 10(b). The braze microstructure appeared fairly uniform up to the edge of the tungsten. EDS analysis of this interface region detected carbon, oxygen, copper, tungsten, and silver, Figure 12.

An x-ray map of a typical area of the braze joint is displayed in Figure 13. The first image is a backscattered electron image of the mapped area, and the following images are x-ray maps for nitrogen, copper, tungsten, silver, and titanium. Nitrogen was concentrated in the core of the dark islands. The copper was concentrated in the layers around the core. Tungsten was detected only for the base metal. Silver was concentrated all around the dark blocky islands. Titanium was highly concentrated at the core of the dark blocky islands; less concentrated in the layers around the core; and highly concentrated at the interface with the ceramic.

## SAMPLE DISPOSITION AND DATA STORAGE

The samples from this project will be returned with this report. All data will be kept on file, and additional report copies can be obtained upon request.

Submitted By:



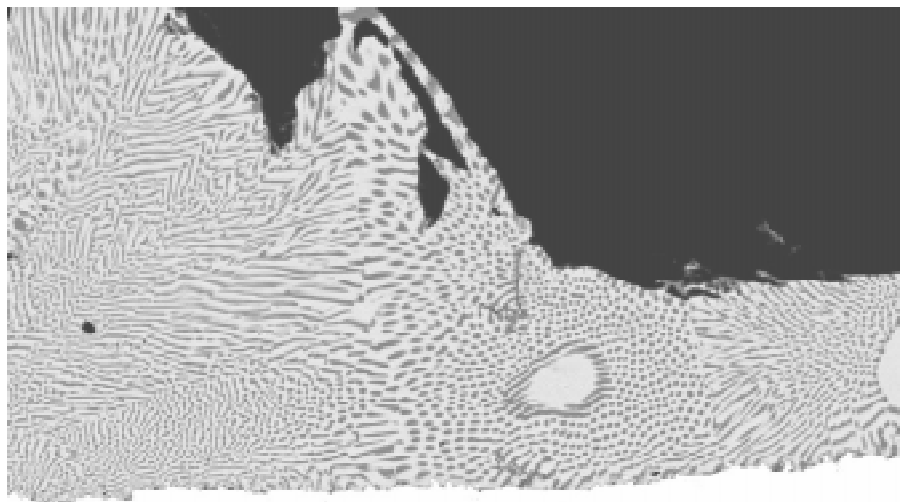
Dieter Scholz  
Laboratory Technologist



Backscattered Electron Image

Magnification: 95X

(a)

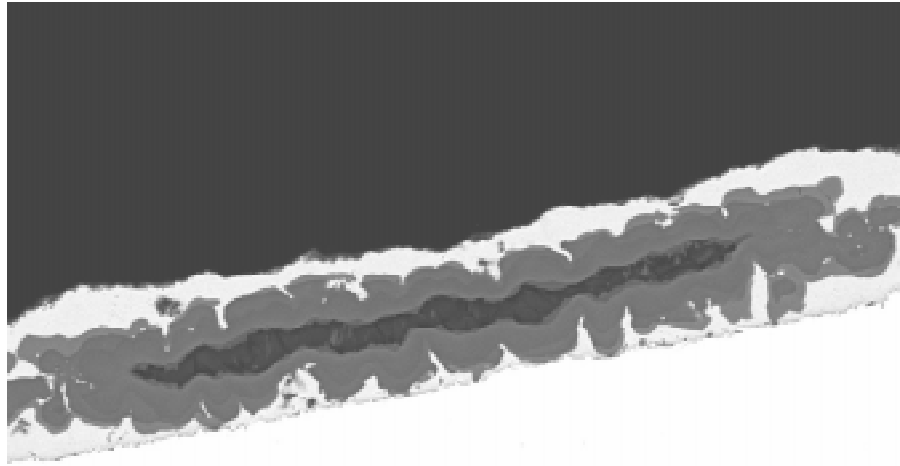


Backscattered Electron Image

Magnification: 1000X

(b)

Figure 1 Typical areas of braze joint microstructure.

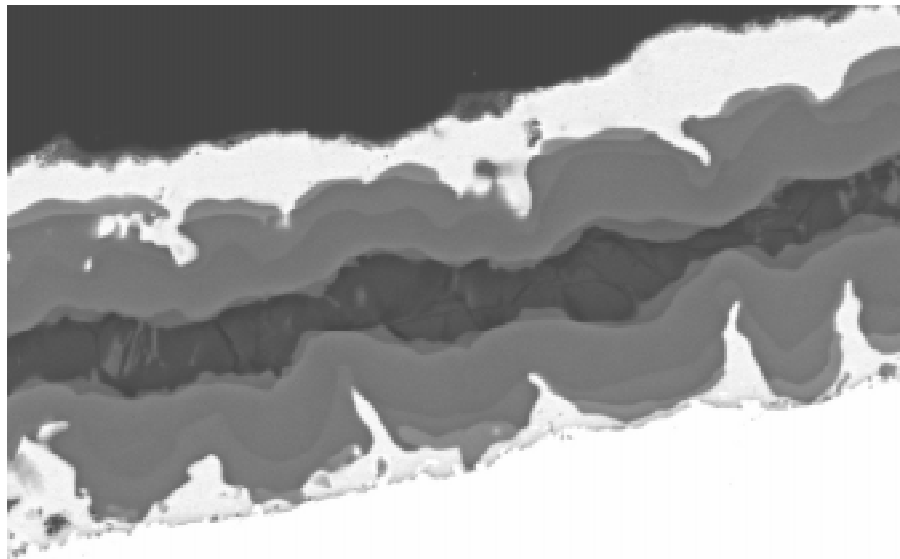


10  $\mu\text{m}$

Backscattered Electron Image

Magnification: 1000X

(a)



10  $\mu\text{m}$

Backscattered Electron Image

Magnification: 2500X

(b)

Figure 2 Typical areas of braze joint microstructure.

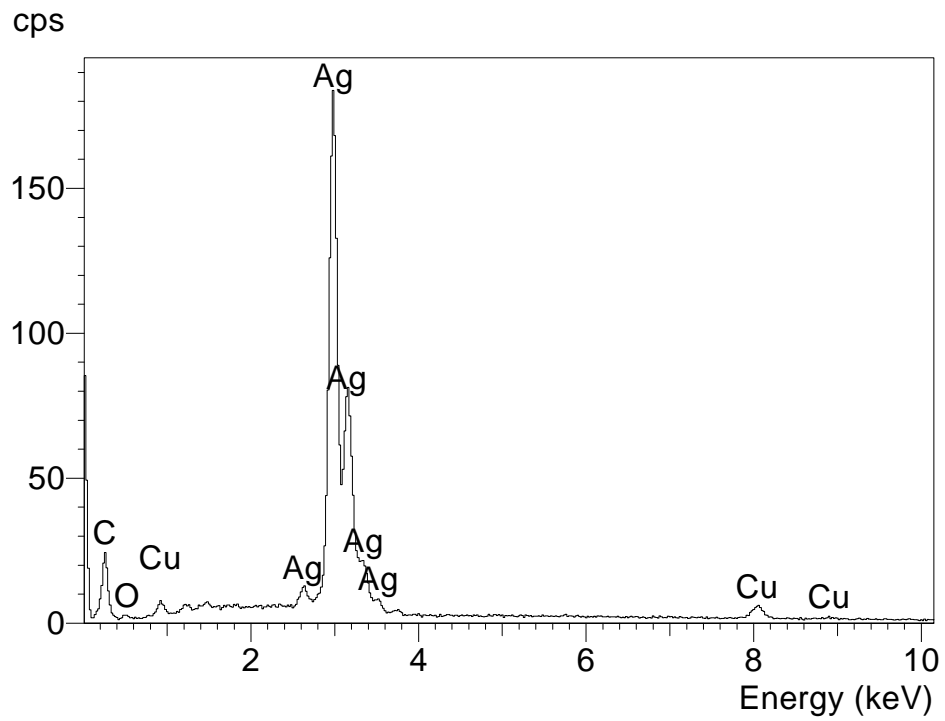


Figure 3 Spectrum for EDS analysis of braze phase A.

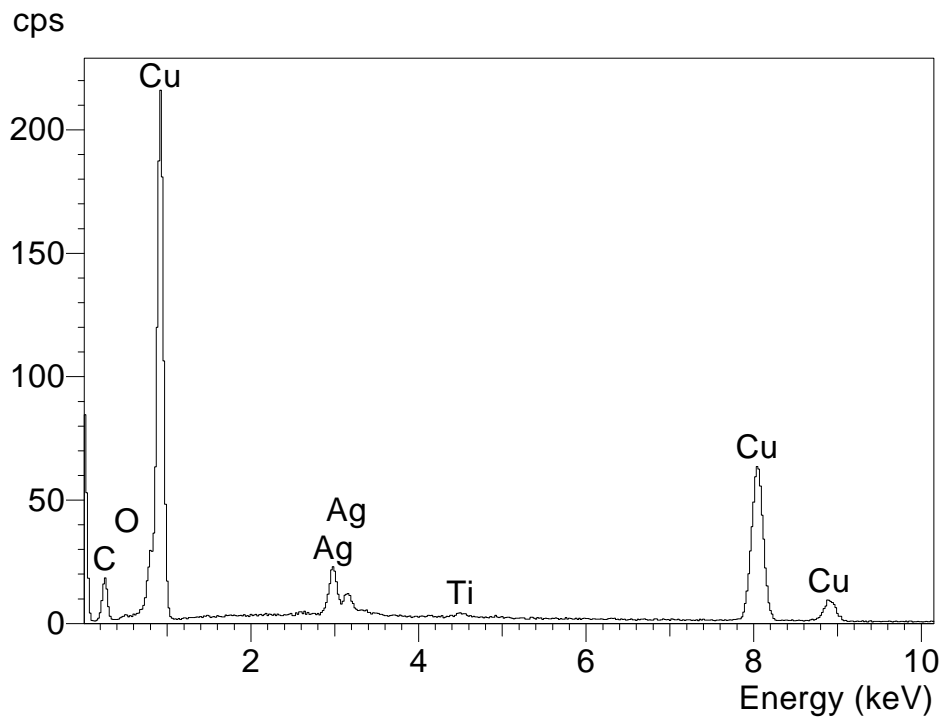


Figure 4 Spectrum for EDS analysis of braze phase B.

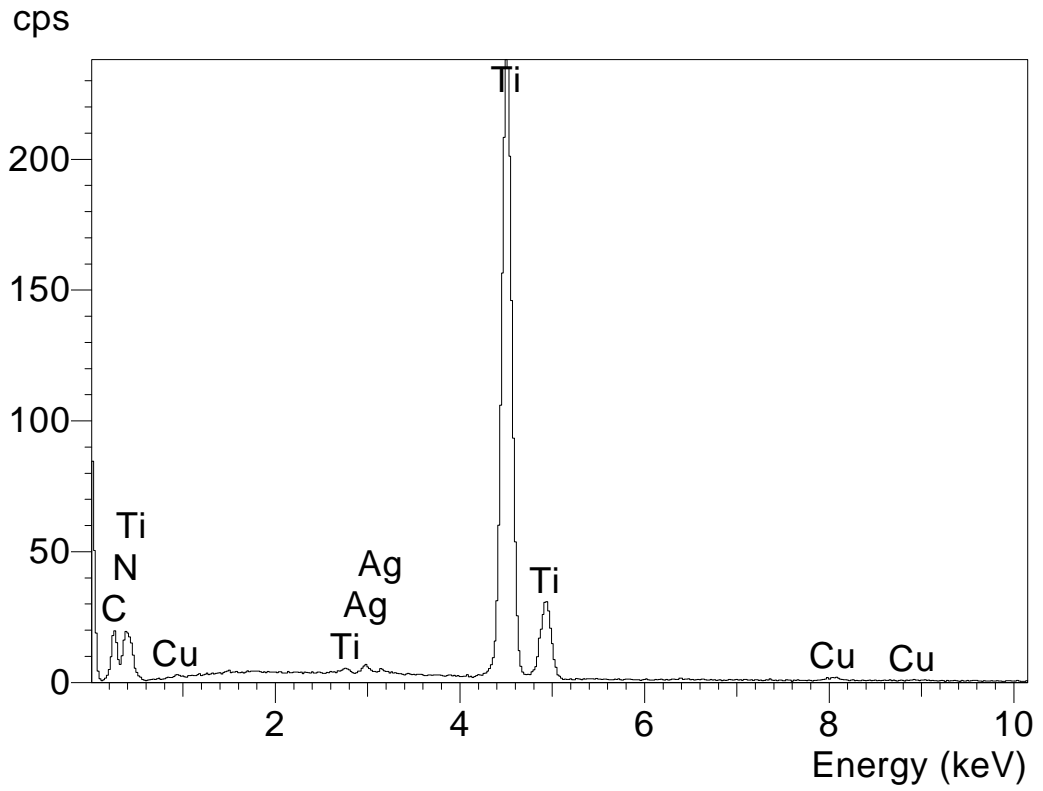


Figure 5 Spectrum for EDS analysis of dark blocky island core.

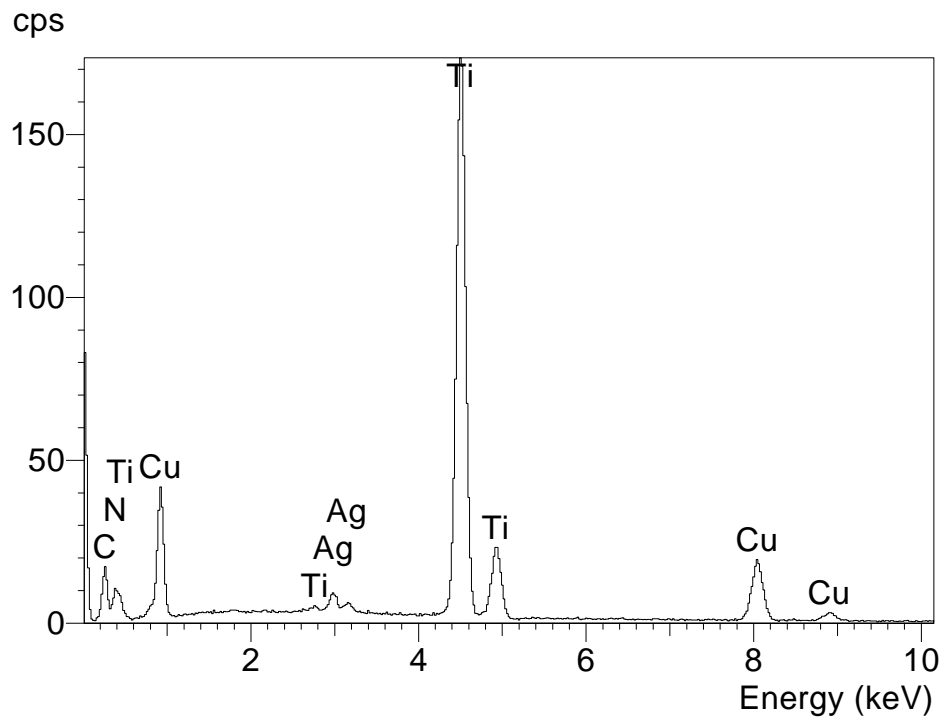


Figure 6 Spectrum for EDS analysis of dark blocky island layer 1.

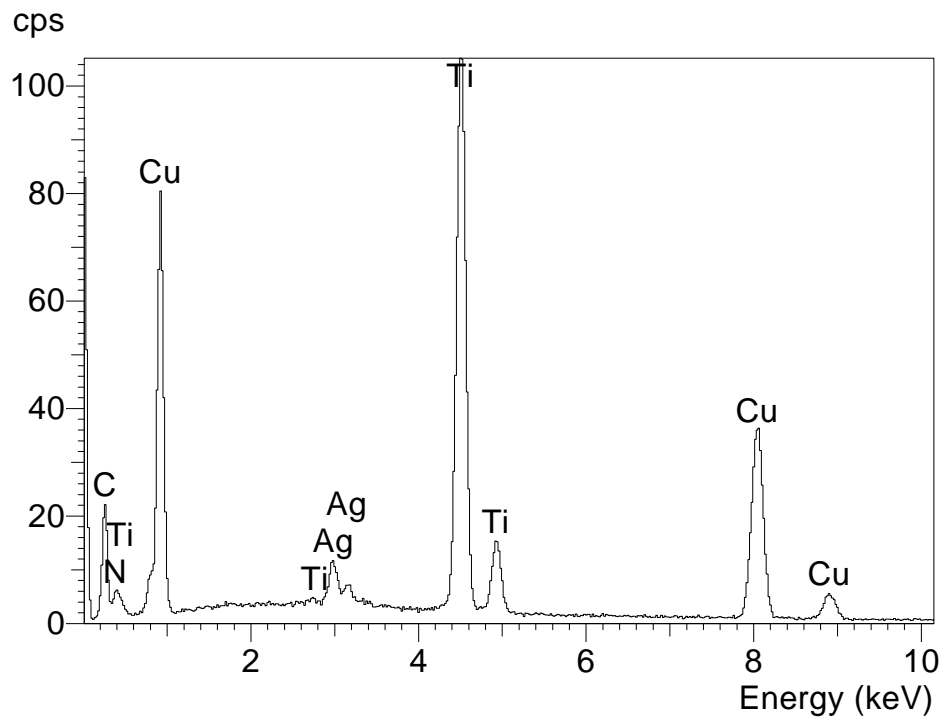


Figure 7 Spectrum for EDS analysis of dark blocky island layer 2.

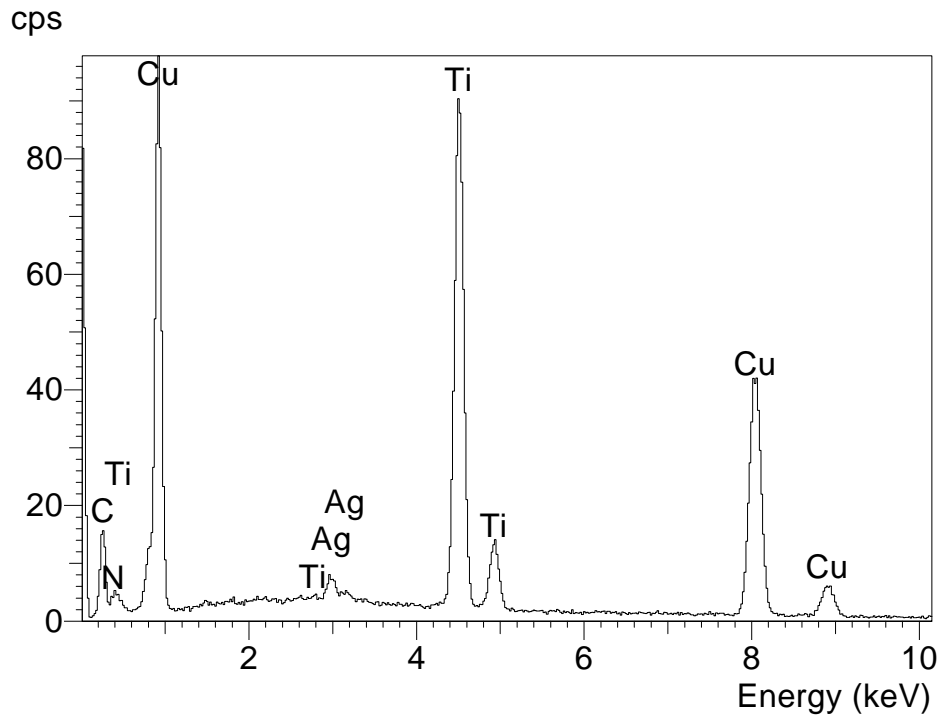


Figure 8 Spectrum for EDS analysis of dark blocky island layer 3.

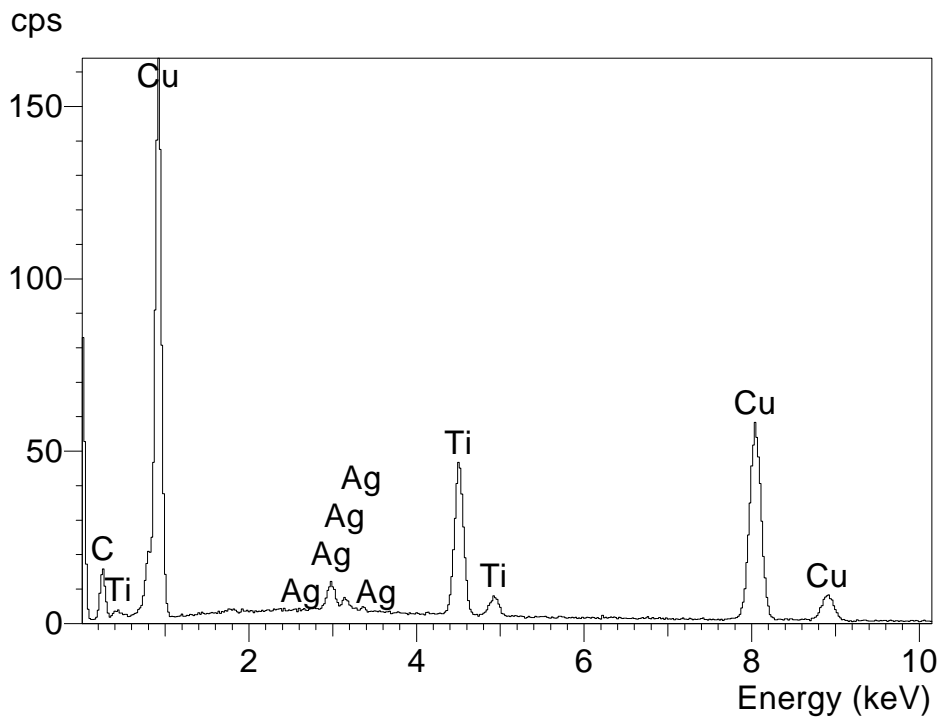
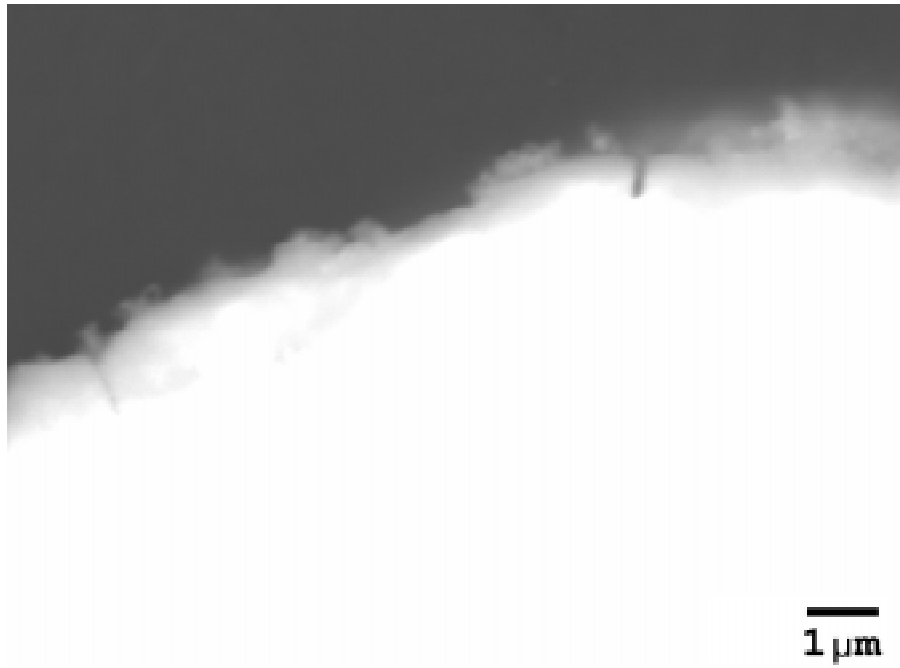


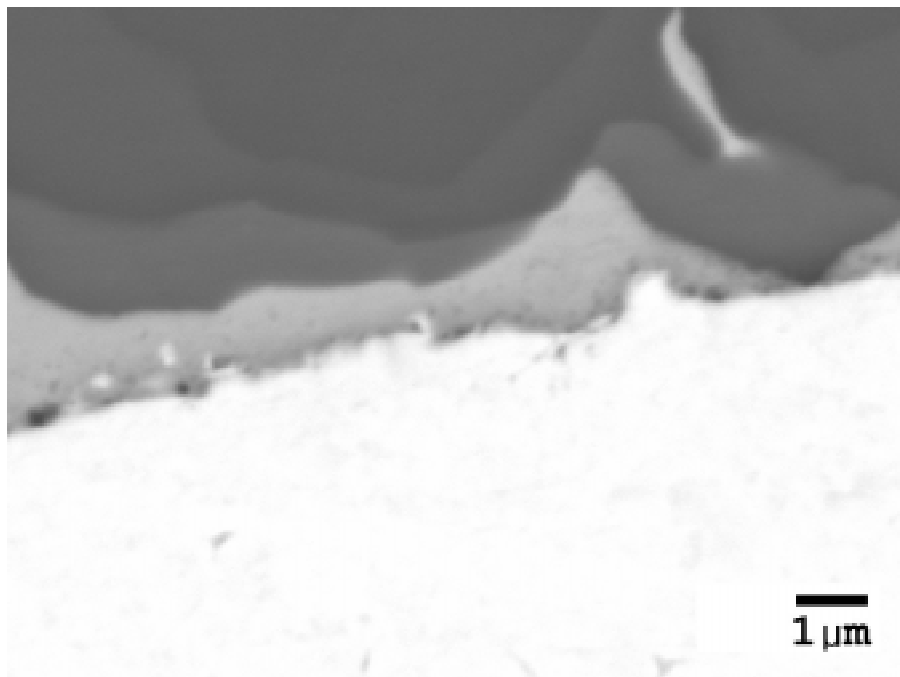
Figure 9 Spectrum for EDS analysis of dark blocky island layer 4.



Backscattered Electron Image

Magnification: 10,000X

(a) Braze/ceramic interface



Backscattered Electron Image

Magnification: 10,000X

(b) Braze/tungsten interface

Figure 10 Microstructure at braze joint interfaces.

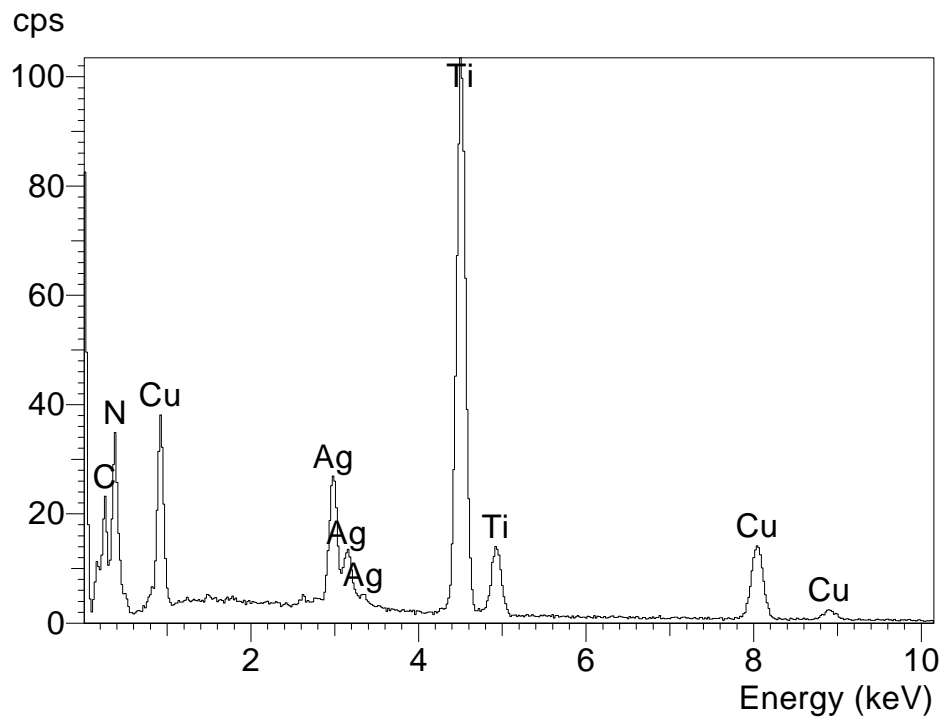


Figure 11 Spectrum for EDS analysis of braze/ceramic interface layer.

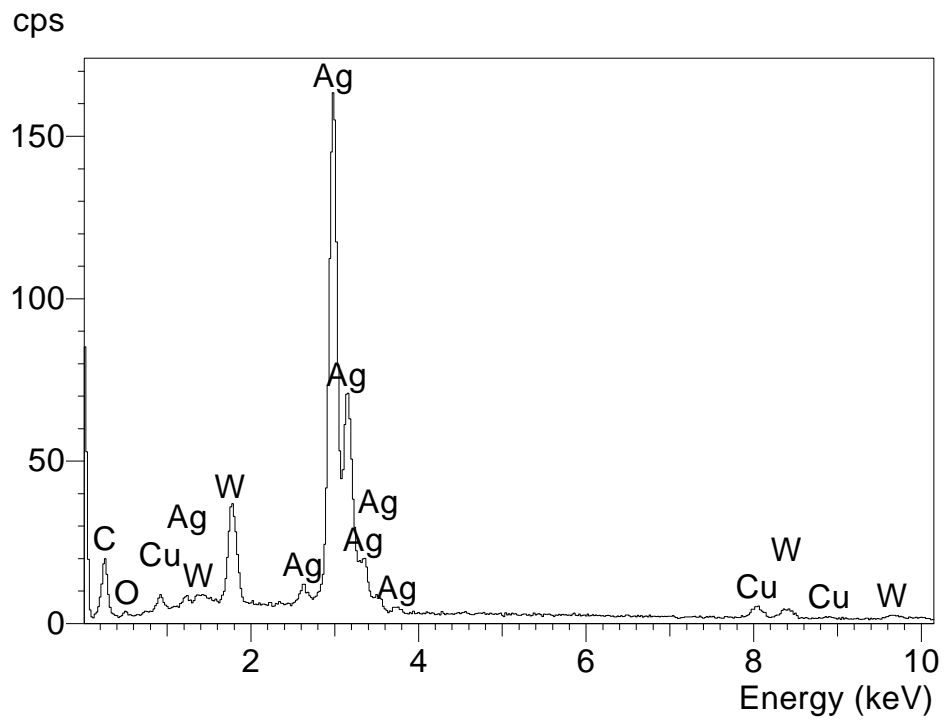


Figure 12 Spectrum for EDS analysis of braze/tungsten interface area.

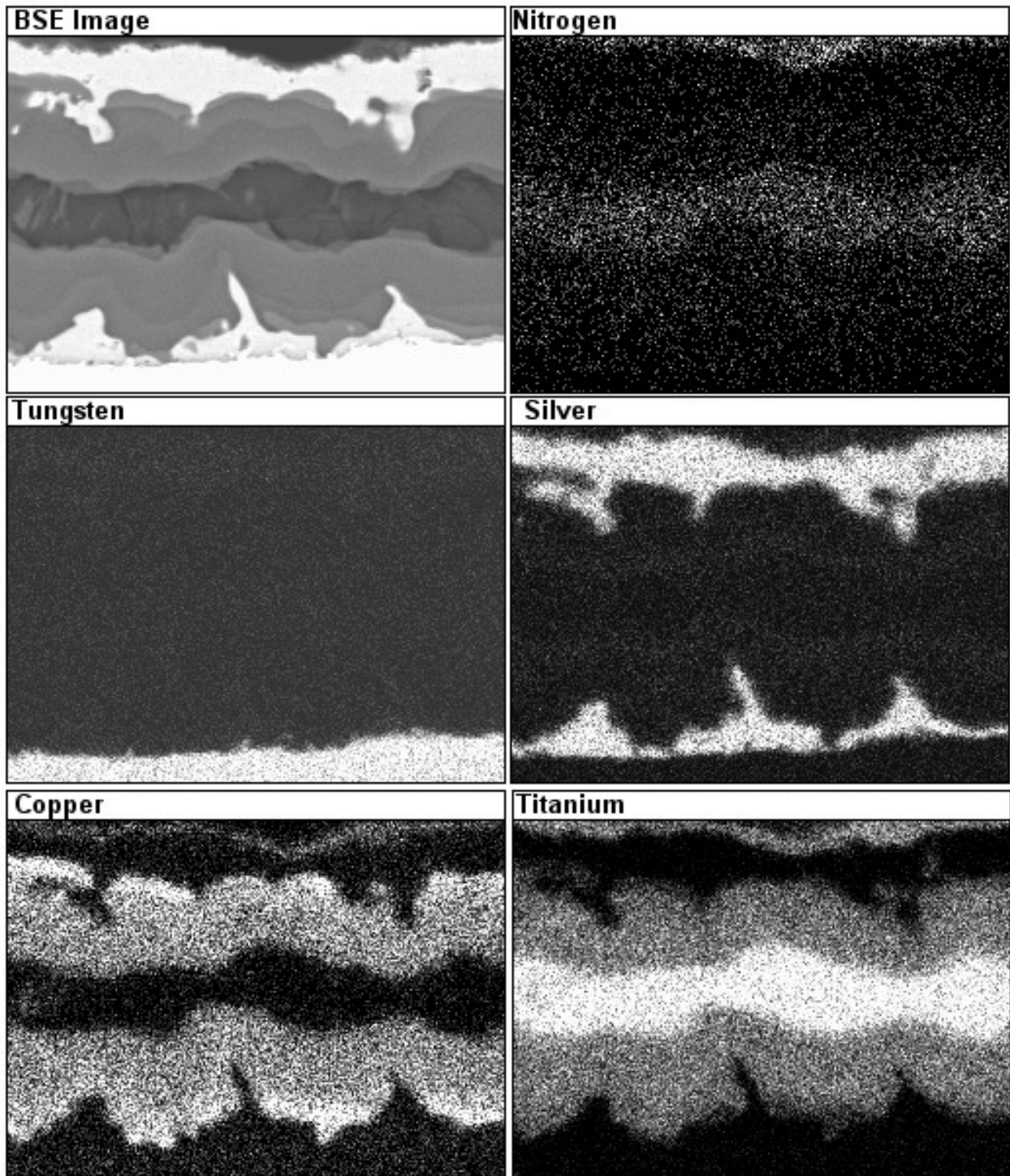


Figure 13 X-ray maps for typical region of ticusil braze joint.