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Addressing Machining Issues for the Intermetallic Compound 60-NITINOL

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Abstract

60-NITINOL (60 wt.% Ni – 40 wt.% Ti) is being studied as a material for advanced aerospace components. Frequent wire breakage during electrical-discharge machining of this material was investigated. The studied material was fabricated from hot isostatically pressed 60-NITINOL powder obtained through a commercial source. Bulk chemical analysis of the material showed that the composition was nominal but had relatively high levels of certain impurities, including Al and O. It was later determined that Al_2O_3 particles had contaminated the material during the hot isostatic pressing procedure and that these particles were the most likely cause of the wire breakage. The results of this investigation highlight the importance of material cleanliness to its further implementation.

Introduction

Recent research and development at NASA Glenn Research Center in conjunction with industrial partners has identified a combination of properties possessed by 60-NITINOL that make this material an excellent candidate for advanced bearings and gears (Refs. 1 and 2). Like its well-known cousin, 55-NITINOL (Ref. 3), this intermetallic material also displays the shape memory effect. Among other properties, 60-NITINOL has excellent aqueous corrosion resistance, is non-magnetic and has a modulus nearly half that of typical steels used in bearing and gear applications.

60-NITINOL is an ordered intermetallic with the CsCl-type B2 crystal structure. In this arrangement, atoms of one type (i.e., Ni) are at the eight corners of a cubic unit cell and an atom of another type (i.e., Ti) sits at the center of the cube, as depicted schematically in Figure 1. Though composed of metallic atoms, the high degree of atomic ordering in this material causes it to behave more like a covalently bonded ceramic with intrinsic brittleness. However, this atomic structure also enables precipitation hardening that results in hardness equivalent to that of conventional bearing materials. This collection of unique properties gives this material a myriad of potential aerospace applications never before explored with intermetallic materials.

During the preparation of prototype 60-NITINOL test components, electrical-discharge machining wires broke repeatedly leading to a great deal of machining downtime. This paper discusses how this issue was addressed.

Materials and Procedures

Gas atomized intermetallic 60-NITINOL powder was obtained from a commercial source. The powder was -60 mesh, as classified by screening. A sample of the powder was examined by field-effect scanning electron microscopy (SEM) and by optical microscopy. The chemical composition of the powder was determined by inductively coupled plasma optical emission spectrometry (ICP-OES). The crystalline phases present in the powder were identified by x-ray diffraction (XRD) using Cu Kα radiation. The apparent density and the tapped density were measured using standard test methods (Refs. 4 and 5). The gravity-driven flow rate of the powder was measured using a Hall flowmeter (Ref. 6). Particle size analysis was performed using the light scattering technique (Ref. 7).

The powder was consolidated into a compact by hot isostatic pressing (HIPping) as depicted in Figure 2. After HIPping, the cylindrical stainless steel container was machined away from the compact and the material was sectioned diametrally into slices approximately 6 mm thick. Blanks for inner and outer bearing races were subsequently sectioned from these slices. All of the machining was done by wire electrical-discharge machining (EDM).

The EDM wire broke several times during the machining of this material. Careful observation after one occurrence revealed a small irregularity in the material where the wire broke. Therefore, this irregularity was suspected to have a role in the EDM wire breakage. Remnants of the material were

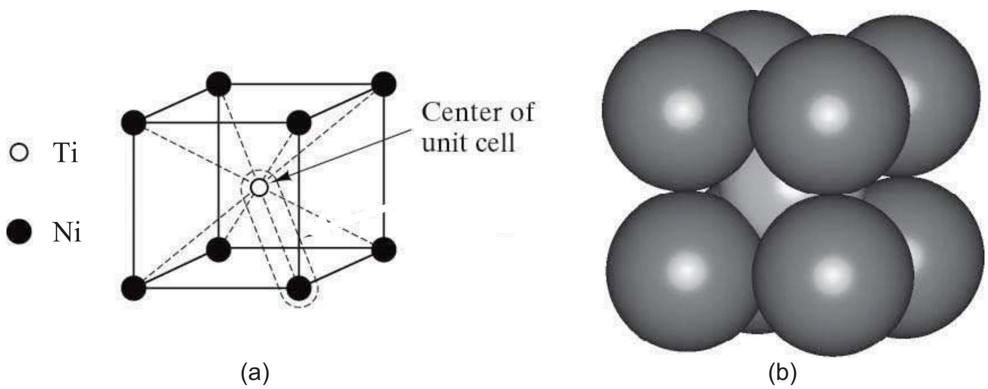


Figure 1.—60-NITINOL B2 unit cell showing (a) atomic arrangement and (b) relative size of atoms in the unit cell (Image generated with Materials Studio modeling package from Accelrys Software, Inc.).

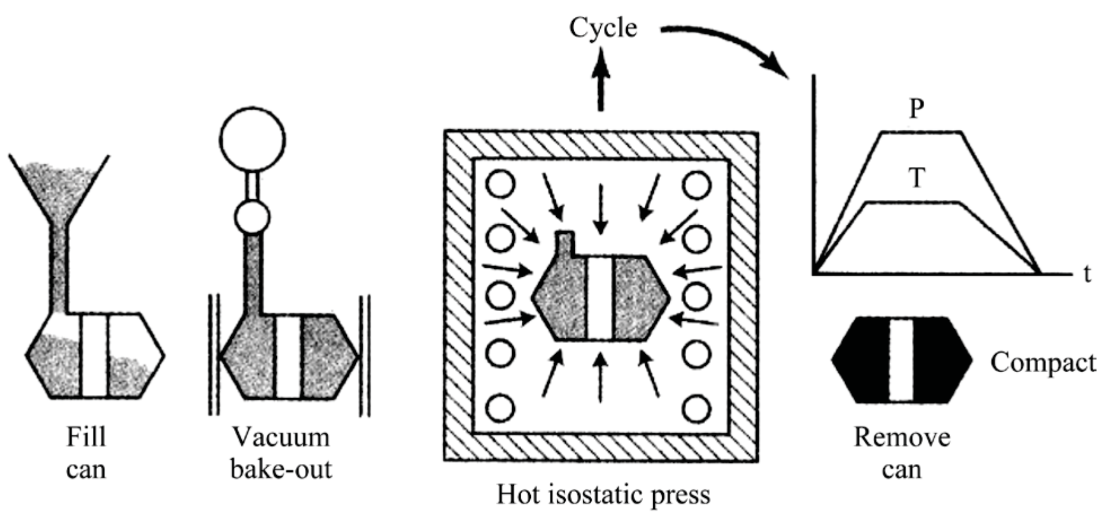


Figure 2.—Schematic description of the steps involved in the hot isostatic pressing (HIPping) process (Ref. 8).

sectioned and prepared for metallographic examination and microindentation hardness testing. Chemical analysis of the bulk material was performed by ICP-OES. Phase identification was performed by energy-dispersive x-ray spectroscopy (EDS) and by XRD.

Results and Discussion

Scanning electron photomicrographs of the 60-NITINOL powder are shown in Figure 3. The generally spherical shape of the powder is typical of gas atomized powders. Some larger particles have satellites from where smaller particles impacted with them before they were completely solidified (see Figure 3(b)). There is also a small portion of irregularly shaped particles within the powder, which is not abnormal with this powder fabrication technique, though these particles are typically removed during the screening step. Images of powder particle cross-sections are shown in Figure 4. The remaining powder characterization data is listed in Table I. One important item to note is the relatively high level of oxygen, iron, aluminum and copper impurities.

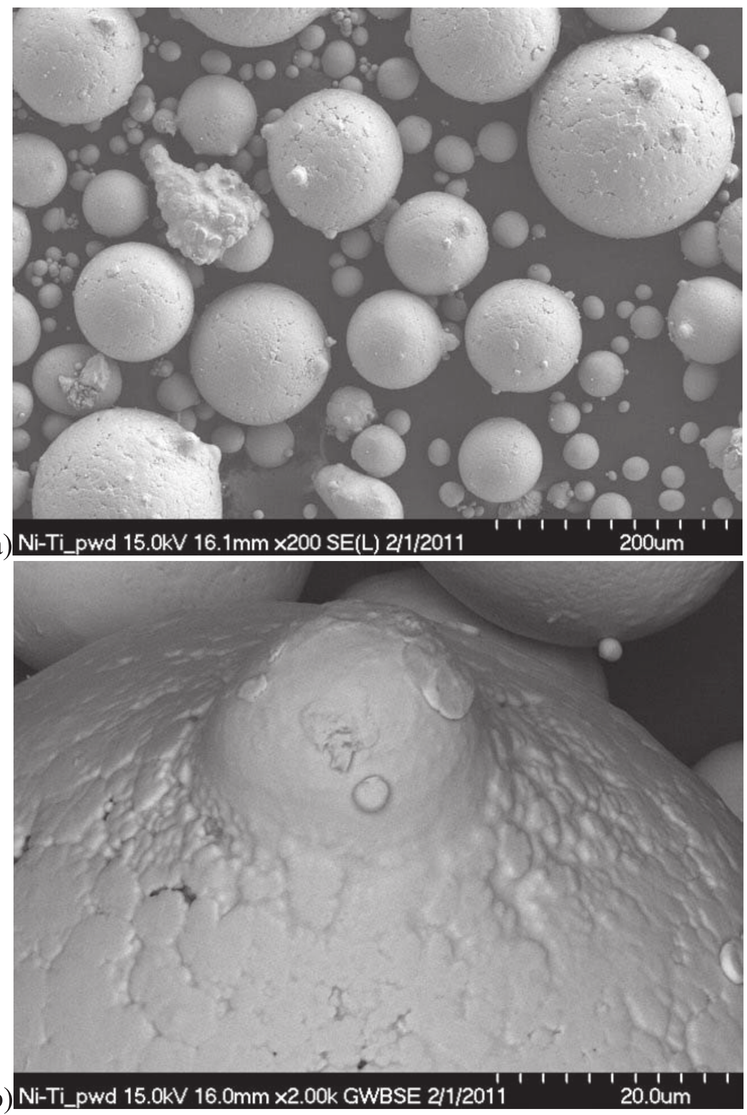


Figure 3.—Scanning electron photomicrograph of the 60-NITINOL powder characterized in Table I shown at (a) 200X original magnification and (b) 2,000X original magnification.

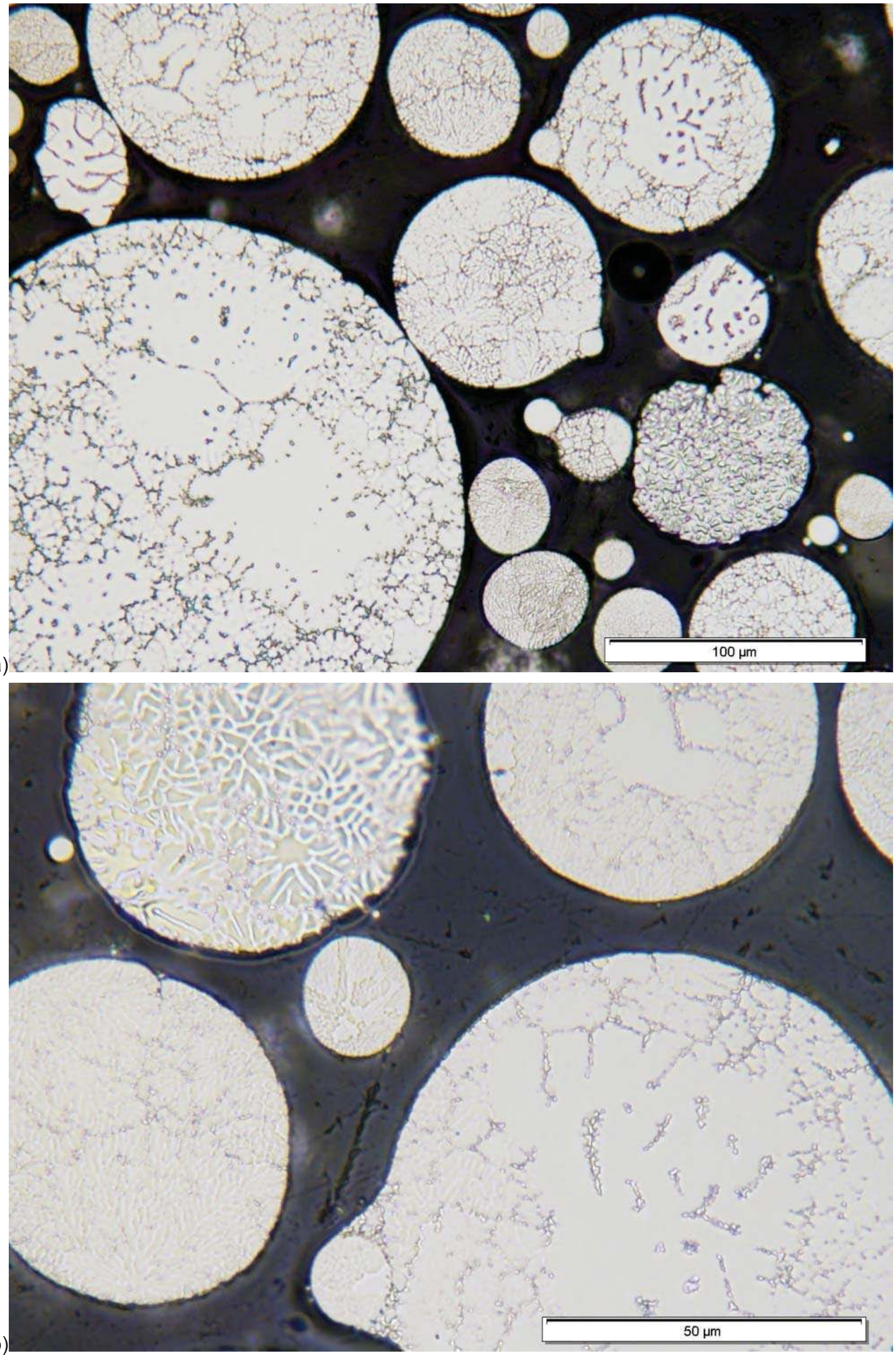


Figure 4.—(a) Optical photomicrographs of cross-sections of the 60-NITINOL characterized in Table I shown and (b) a higher-magnification image. The polished specimen was swab-etched with a room temperature solution composed of $1HF+4HNO_3+5H_2O$.

TABLE I.—CHARACTERIZATION OF 60-NITINOL POWDER SHOWN IN FIGURE 3 AND FIGURE 4

Composition	59.7wt%Ni-40.1wt%Ti
Impurities (ppm)	O (655), Fe (480), Al (230), Cu (45), Cr (30), Co (20), V (20)
Crystalline phases	47% Cubic (B2) NiTi 53% Rhombohedral ($D0_{24}$) Ni_3Ti
Apparent density	4.0 ± 0.0 g/cm^3 (70%*)
Tap density	4.9 ± 0.0 g/cm^3 (86%*)
Hall flow time (50g sample)	16.6 ± 0.4 sec
Particle size	D_{mean} = 100.5 ± 60.7 µm D_5 = 21.5 µm D_{10} = 30.7 µm D_{50} = 94.0 µm D_{90} = 178.7 µm D_{95} = 201.7 µm

*The density is expressed as a percentage of the theoretical density of B2 NiTi (6.556 g/cm^3), based on x-ray powder diffraction data.

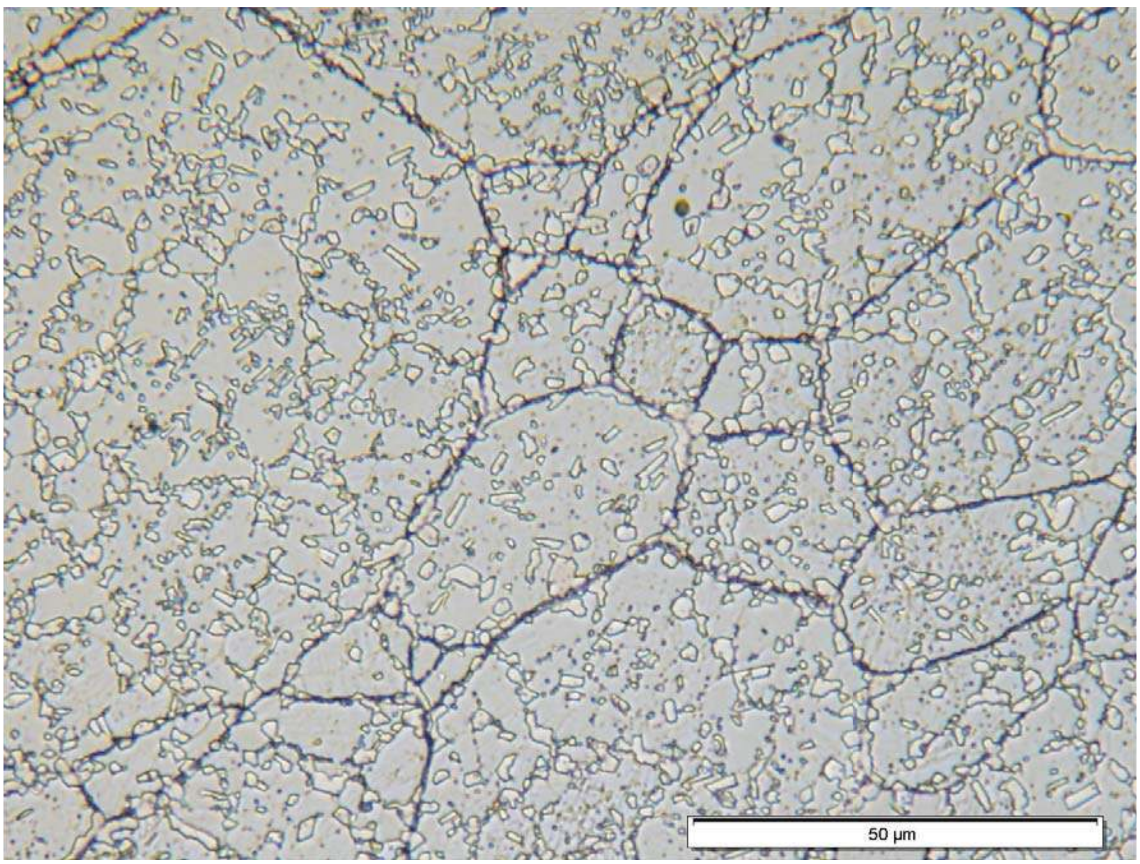


Figure 5.—Optical photomicrograph of 60-NITINOL compact after hot isostatic pressing. The polished specimen was swab-etched for 20 seconds with a room temperature aqueous solution composed of 10 vol.% HF and 5 vol.% HNO_3.

XRD determined that the two phases present in the compact are cubic NiTi and orthorhombic Ni_3Ti. The microstructure of the as-received HIPped 60-NITINOL material is shown in Figure 5. EDS was used to confirm that the parent phase is NiTi and the light grey second phase dispersed throughout the microstructure is Ni_3Ti. The grain boundaries represent the prior particle boundaries. An SEM photomicrograph and an elemental dot mapping of the 60-NITINOL compact near the stainless steel HIPping can are shown in Figure 6. The can is visible along the left of the image. These images merely help to confirm that the composition of the material is uniform within the compact. Additional compact characterization data, including the bulk chemical composition, is listed in Table II. Again, the levels of oxygen, iron, aluminum and copper (detected at levels on the order of hundreds of parts per million) should be noted.

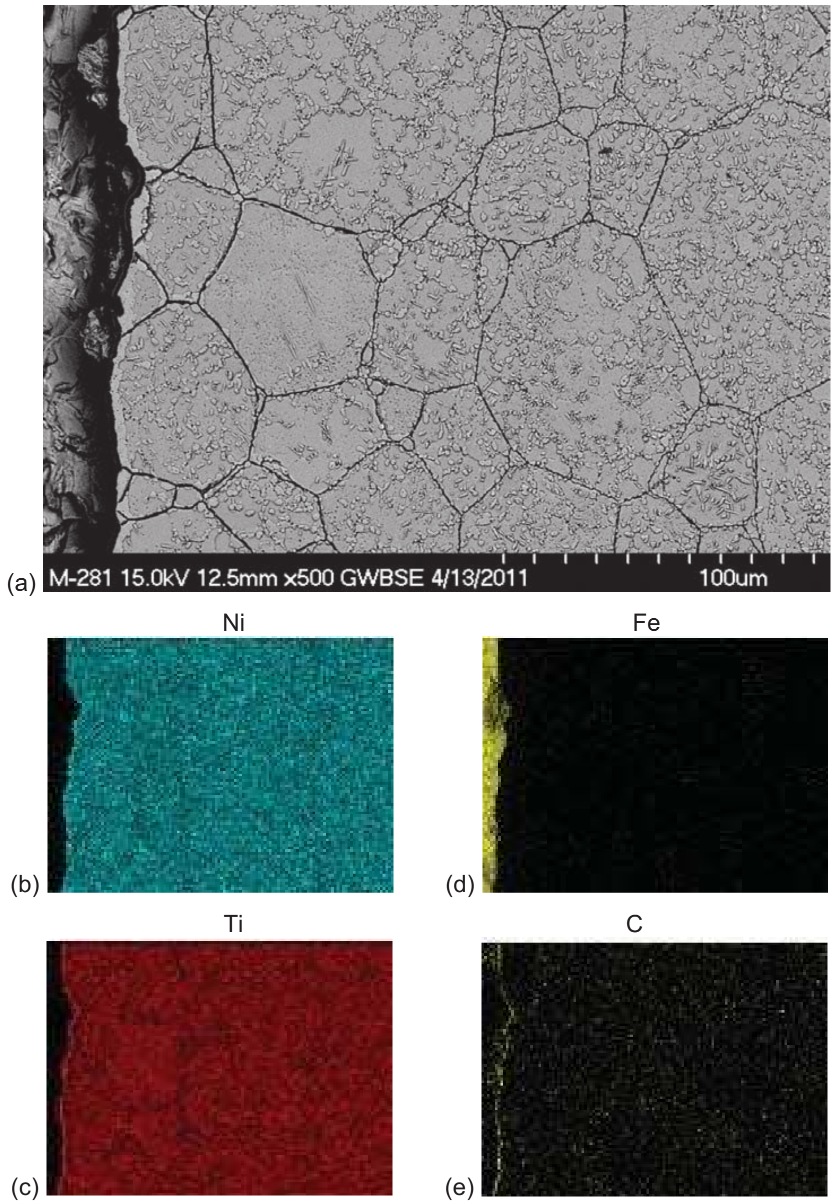


Figure 6.—(a) SEM photomicrograph of the 60-NITINOL compact near the stainless steel container (seen on the left of image) and (b) to (e) x-ray elemental dot maps from this image showing the major elements present.

TABLE II.—PHYSICAL PROPERTIES OF THE AS-RECEIVED 60-NITINOL COMPACT

Composition	59.9wt%Ni- 39.9wt%Ti
Impurities (ppm)	O (690), Fe (490), Al (210), Cu (105), Cr (30), Co (20), V (20)
Density	6.645 g/cm^3
Hardness	339.2 ± 9.0 HV$_{500}$

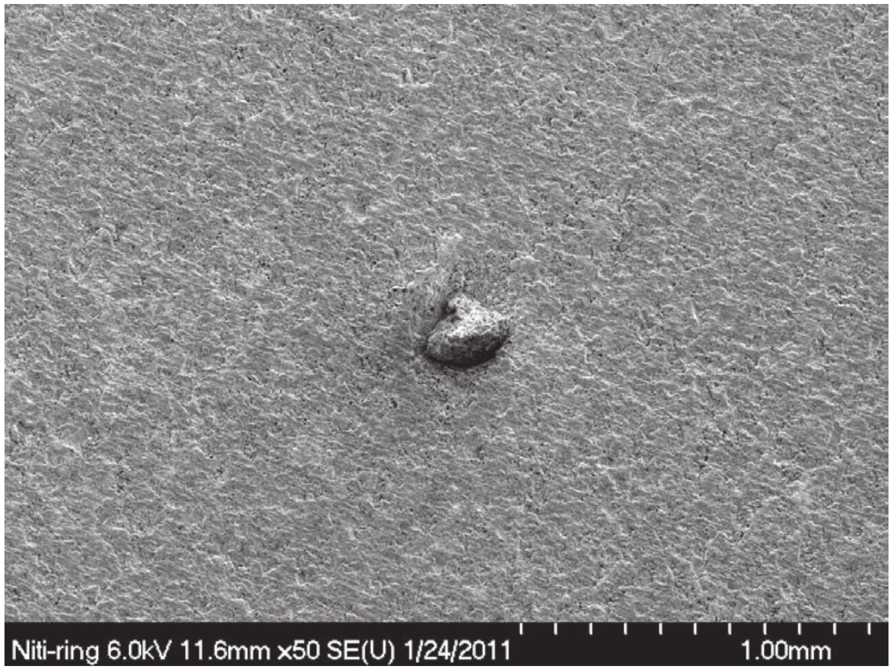


Figure 7.—SEM photomicrograph of an inclusion discovered on the inner diameter of an outer bearing race blank.

Upon closer examination of the irregularity suspected to be involved in an EDM wire breakage, it was identified as a particle embedded in the material (see Figure 7). As shown in Figure 8, the particle was composed of aluminum and oxygen. The ratio of these two elements indicates that the particle is aluminum oxide (Al_2O_3). The particle was also splattered with a small amount of a copper alloy (brass), which was transferred from the EDM wire (see Figure 9).

It was later discovered that an abrasive flap wheel had been used during the procedure used to prepare the HIP container. A sample of the flap wheel abrasive was obtained. The abrasive particles were found to be embedded in an adhesive material and then covered with a coating that allows the abrasive to be exposed gradually during its intended use. The coating was dissolved away with acetone to facilitate SEM analysis of the abrasive particles. Figure 10 shows the abrasive particles partially exposed through the coating. These particles are on the order of hundreds of microns in size and develop static charge when probed with the SEM electron beam. A typical x-ray spectrum of one of these particles (Figure 10(b)) indicates that they are indeed the same composition as the particle embedded in the 60-NITINOL (namely, Al_2O_3). This partially accounts for impurities detected in bulk chemical analyses (i.e., Al and O). Clearly, the EDM wire breakage was caused in this case because the wire was unable to pass through this electrically insulating inclusion. This was most likely the case with most (if not all) of the other wire breakages.

This finding provides an opportunity to highlight an important issue. Due to the brittle nature of intermetallics, material cleanliness is extremely important. It is especially important to prevent ceramic inclusions because they tend to be hard and faceted, which makes them excellent crack initiators. Any tensile stress near such an inclusion could lead to a failure. This issue is discussed in a recent publication that addresses quench cracking of 60-NITINOL due to ZrO_2 contamination (Ref. 9). The alumina contamination studied here was easily eliminated by modifying the HIP container preparation procedure.

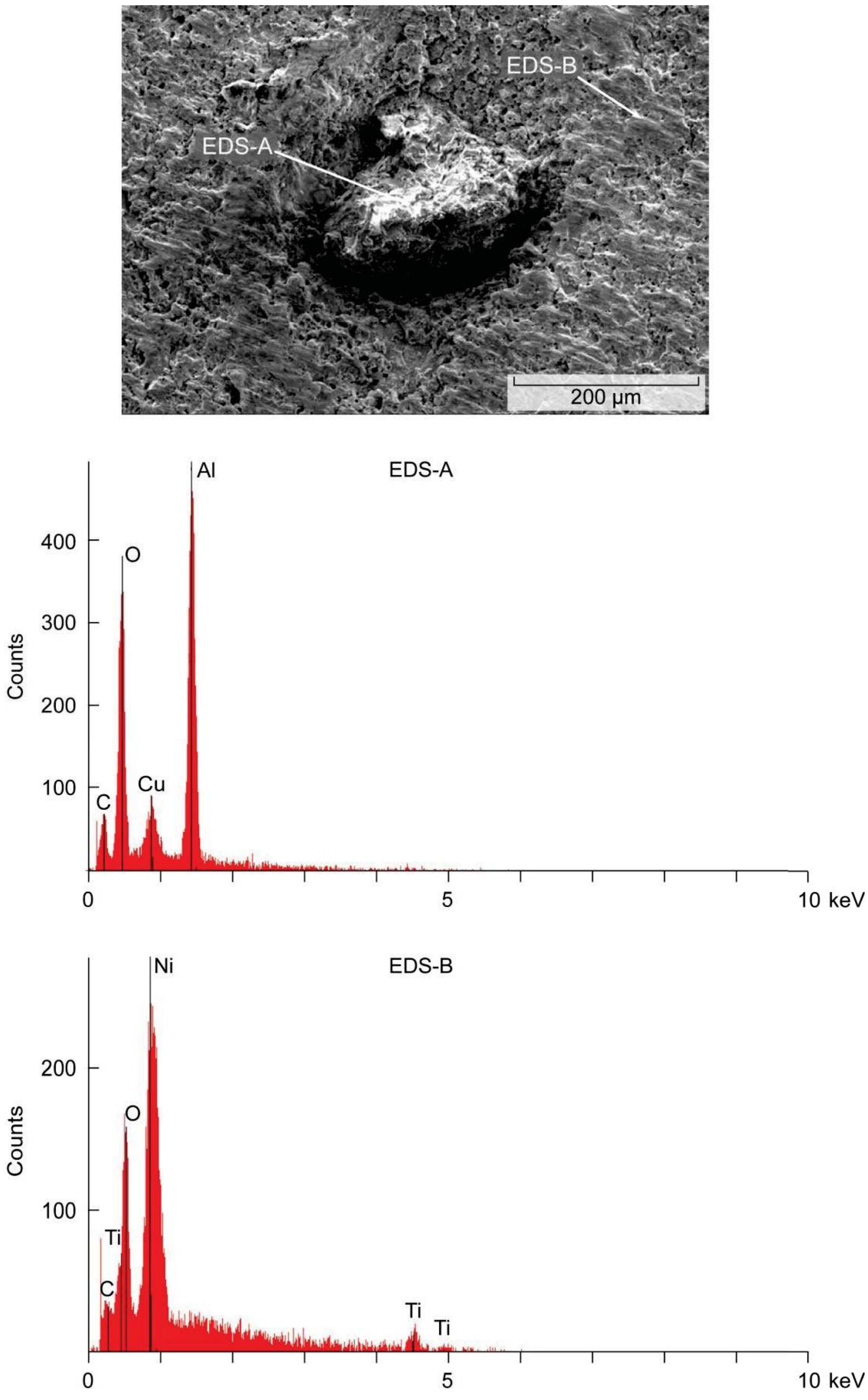


Figure 8.—SEM photomicrograph of the embedded particle. The first x-ray spectrum (EDS-A) indicates that the particle is composed of aluminum and oxygen in a ratio that follows the weight percentage of these elements in alumina. The second spectrum (EDS-B) confirms that the parent phase is 60-NITINOL.

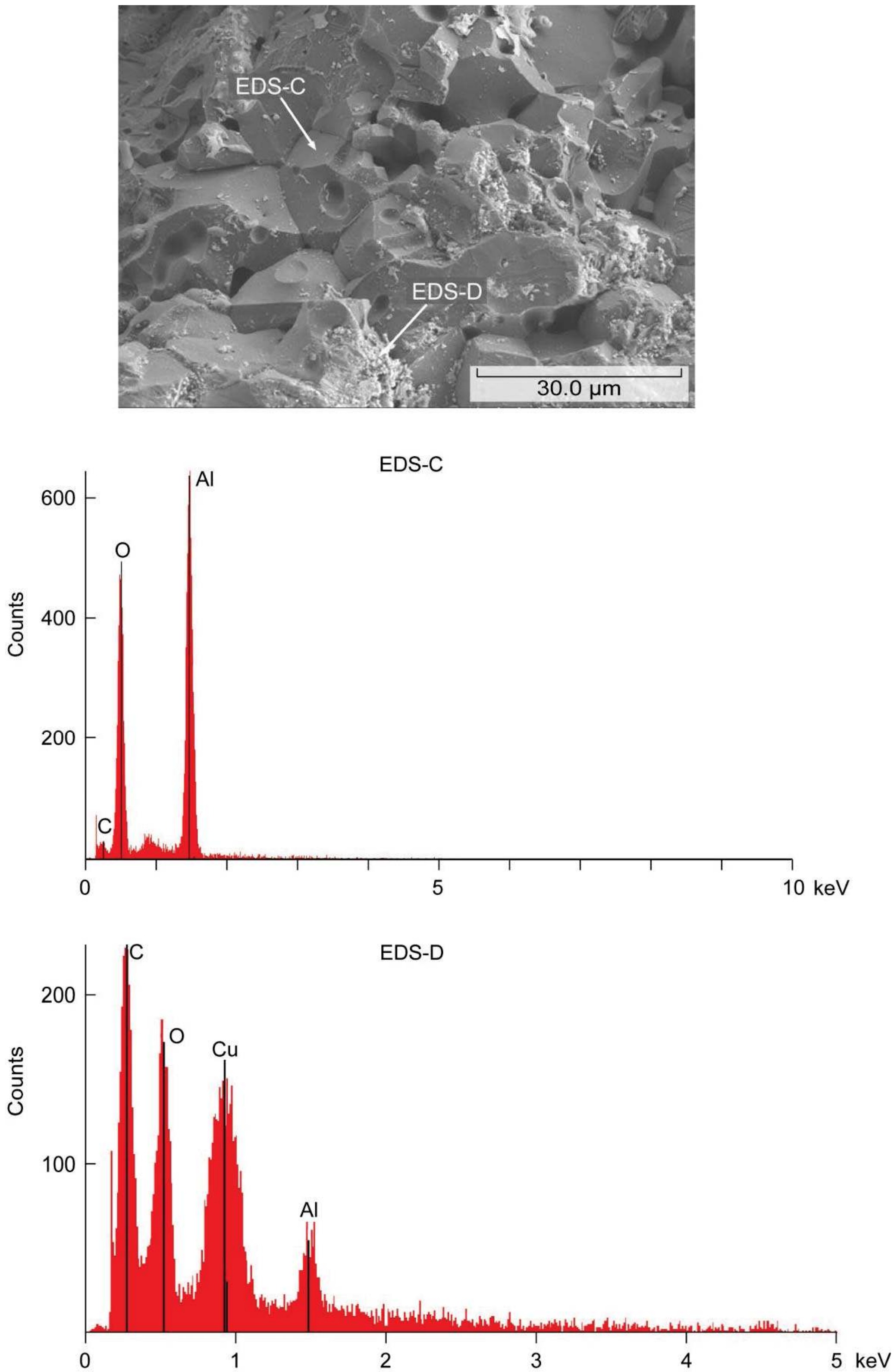
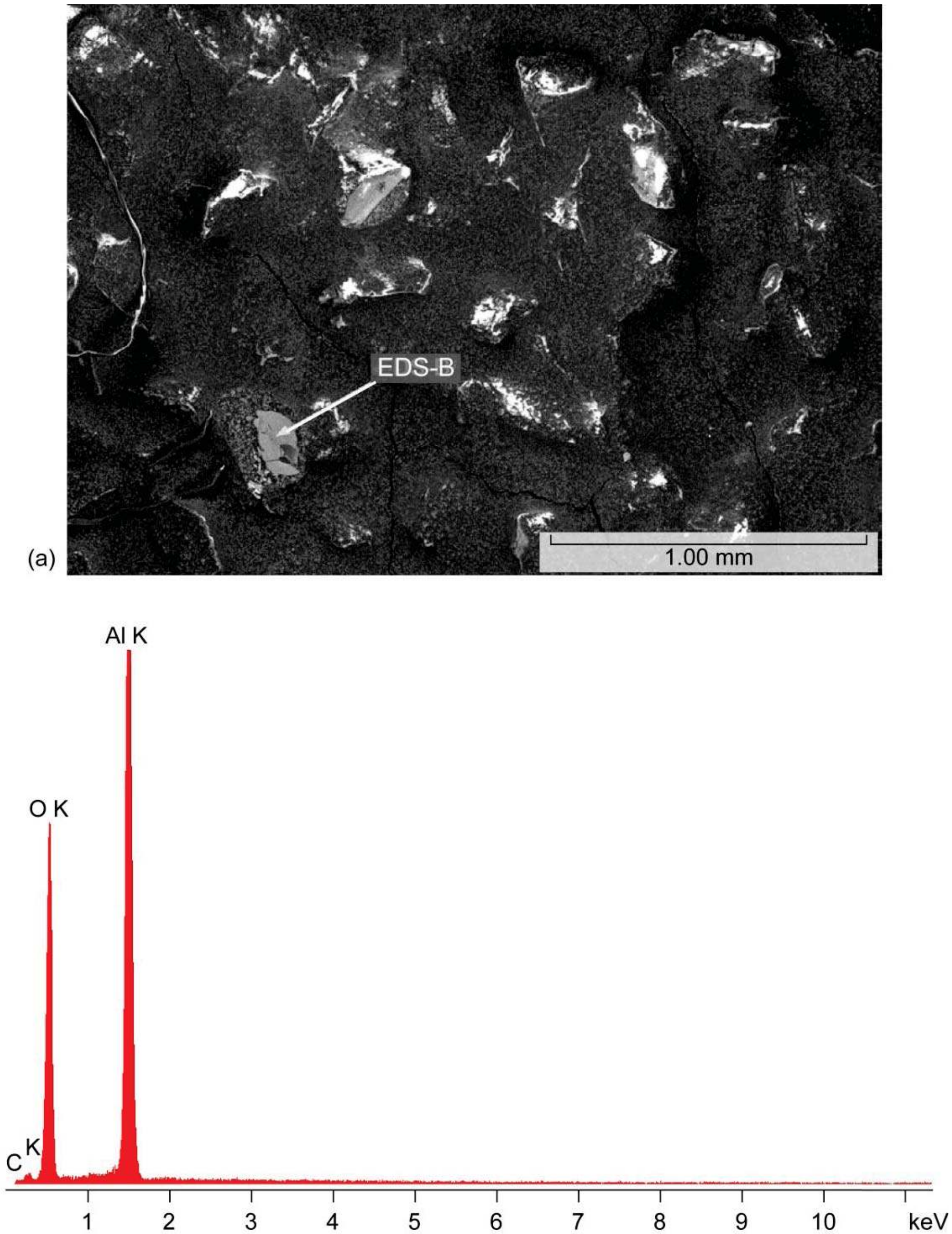


Figure 9.—SEM photomicrograph of surface of embedded particle shown in Figure 8 with EDS spectra indicating a composition of alumina (EDS-C) splatter with brass from the EDM wire (EDS-D).

(a)



(b)

Figure 10.—(a) SEM photomicrograph of surface of flap wheel abrasive paper showing abrasive particles partially exposed and (b) the EDS spectrum of one such particle as indicated in the figure.

Conclusions

The cause of frequent EDM wire breakage during the machining of HIPped 60-NITINOL has been investigated. Based on the results of this investigation, it can be concluded that Al_2O_3 inclusions caused a number of EDM wire breakages during the machining of this material. More generally, any type of ceramic inclusion would tend to degrade material properties such as machinability and structural integrity.

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