Manufacturing and Processing Techniques Affecting Morphology of Pyrotechnic Oxidizer Particles

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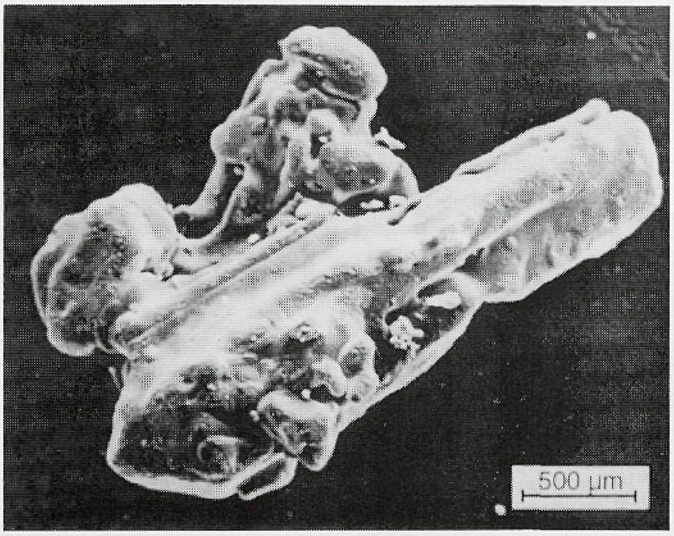
In a pyrotechnic composition, the morphology (size, shape and surface features) of individual particles in the mixture influences both burn rate and the ease of ignition of the composition. Collectively, the larger, more rounded and smoother the particles, the lower the burn rate and the more difficult it is to ignite the composition. However, the purpose of the current investigation was only to catalogue some particle morphologies that may be useful in forensic identification of unconsumed or residual pyrotechnic evidence. In this study, potassium nitrate was chosen because of its common use in pyrotechnics and because of its similarity with other frequently used pyrotechnic oxidizers.

All samples were prepared for scanning electron microscopy using a double-sided adhesive carbon disk applied to an aluminum post. Particles were mounted to the carbon disk and sputter coated with gold. Examination of the particles was performed at Mesa State College using a newly remanufactured AMRAY 1000 and a digital imaging system. An accelerating voltage of 20 kV was used with a final aperture diameter of 200 µm. The working distance was approximately 12 mm.

The primary source of potassium nitrate investigated was analytical reagent (AR) grade with an initial particle size of 250–3400 microns. Another source was an agricultural prill (AgP) with an initial particle size of 100–1000 microns. The AR grade particles initially presented numerous linear structures, whereas the AgP presented mainly spherical structures, see Figure 1.

Material of this size is much too coarse to be useful in pyrotechnics and must be reduced in size. One common method of particle size reduction is by crushing or grinding. Often this is accomplished using a rotating disk mill similar to a grain mill, utilizing either metal or stone disks. The particles shown in Figure 2 are the result of such milling. While the goal was to reduce the particles to a size of less than approximately 300 microns, numerous particles smaller than 20 microns are also produced—seen here *adhering to the surface of the larger particle. (Quite similar* appearing particles are also produced by crushing, using a simple mortar and pestle.)

Another common method of particle size reduction is to em-



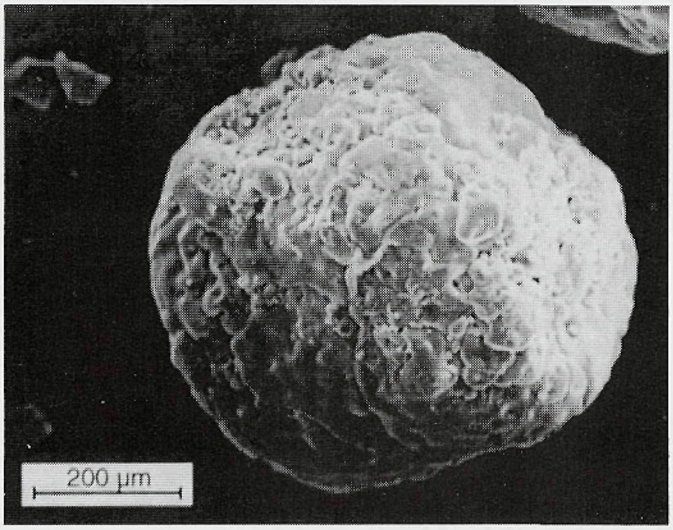
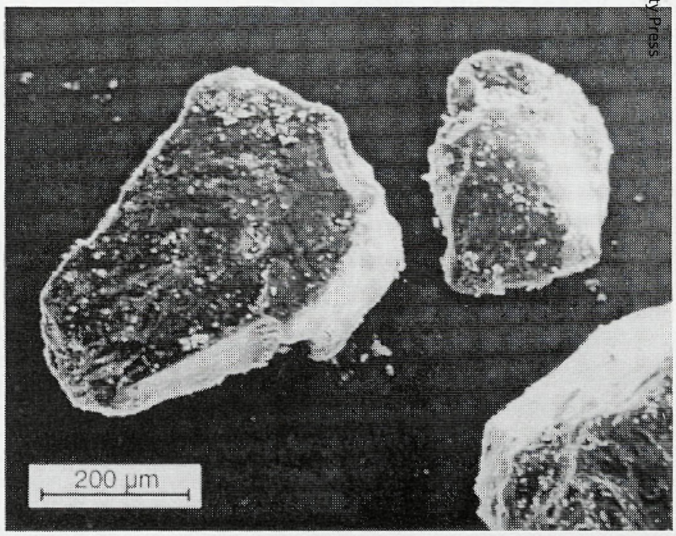


Figure 1. Somewhat typical examples of the AR (upper) and AgP (lower) grade potassium nitrate particles. Upper, 35X magnification, lower, 100x magnification.



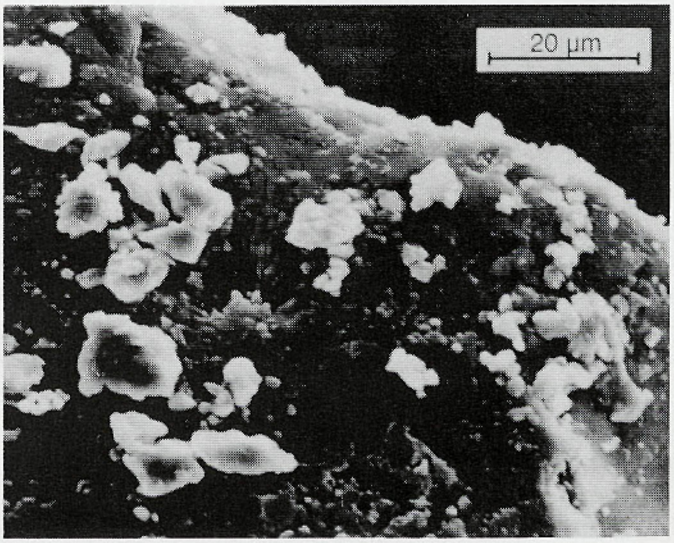


Figure 2. Two images of AR grade potassium nitrate, 60 to 100 mesh particles produced using a rotating disk mill, documenting the presence of many tiny particles adhering to the surfaces of larger particles. Upper, 100x magnification, lower 1000x magnification.

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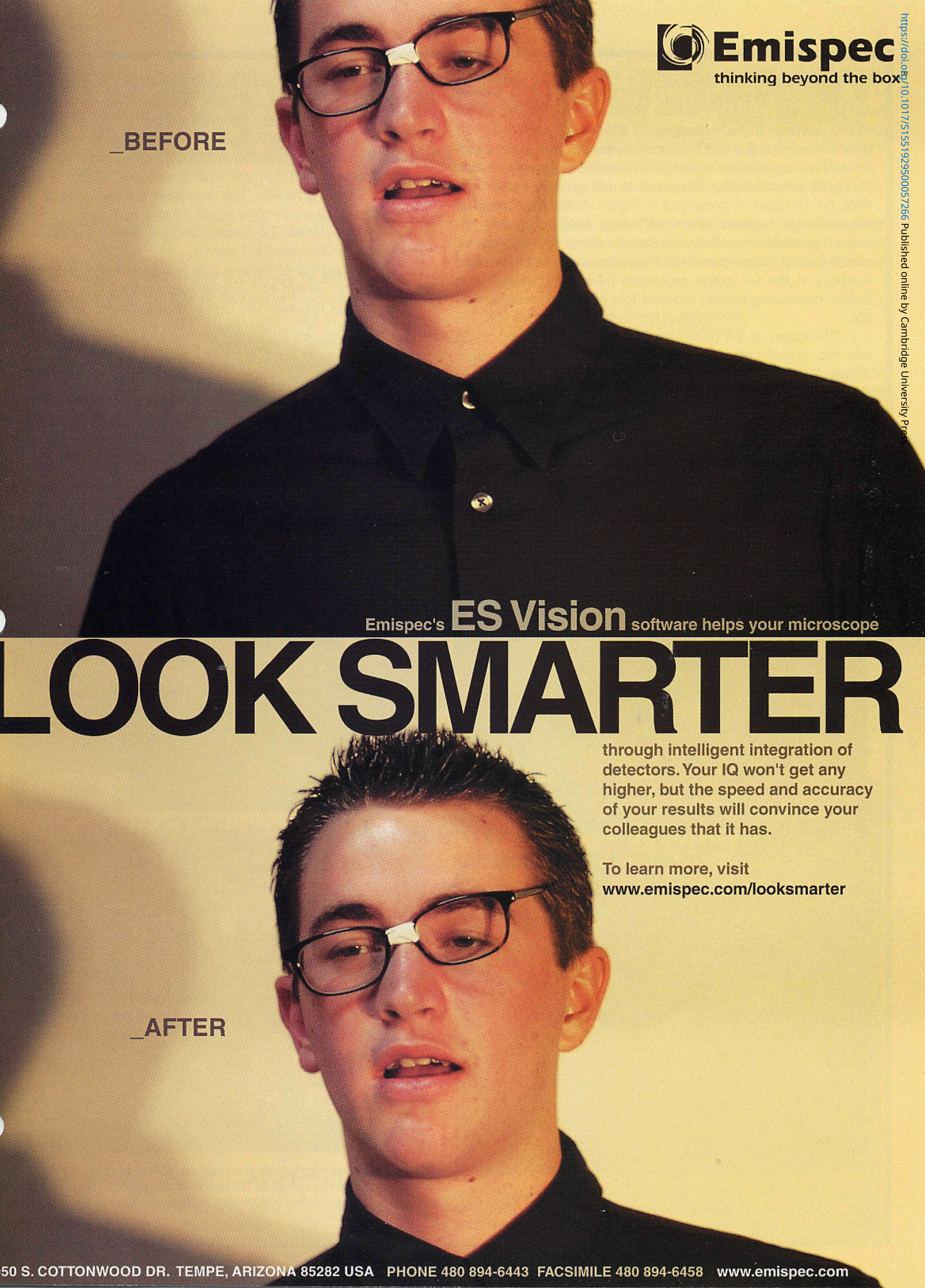
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ploy the use of a ball mill. In this case, the ball mill consisted of a rotating drum containing ceramic cylinders (12 mm in diameter by 12 mm in length). The result of milling the potassium nitrate for 12 hours is presented in Figure 3. Although most of the individual particles are significantly smaller than 40 microns, there was a tendency for them to clump or fuse together creating much larger particles. This was due to slight residual moisture in the AR grade potassium nitrate; thorough drying before processing greatly reduces this tendency to agglomerate.

The shape of the particles after processing tended to fall into two distinct categories, those with sharp angular features (Figure 2) and those with rounded margins (Figure 3). These categories (sharp and rounded) can be helpful in identifying processing techniques of the materials used in manufacturing pyrotechnic explosive devices. However, perhaps more importantly, such characterization can be useful in establishing a forensic match between known and evidentiary materials.

A third method of particle size reduction—coacervation—has gained some popularity among individuals because it does not require machinery. In this case, potassium nitrate is dissolved in water to create a nearly saturated solution; this is often performed while heating the solution to expedite the process and increase the amount of material in solution. Once the material is in solution, it is removed from the heat source and alcohol is quickly added while stirring rapidly. The potassium nitrate is forced out of solution forming particles of a much smaller particle size than the original material. Their appearance is quite distinct from the other methods and is illustrated in Figure 4.

The last method of particle size reduction to be discussed is hammer milling, where high-speed metal blades are used to reduce the size of the material. These blades shatter the potassium nitrate crystals and produce much more noticeable fracture patterns than crushing or ball milling. Figure 5 illustrates the extensive fracture patterns and whiskering (the more or less parallel ridges on the fractured surface of the crystals).

There are other surface features that may aid in identifying the processing method or in establishing a forensic match. The first are pits or divots on the surface of larger particles such as seen in Figure 6. These particles were produced by ball milling

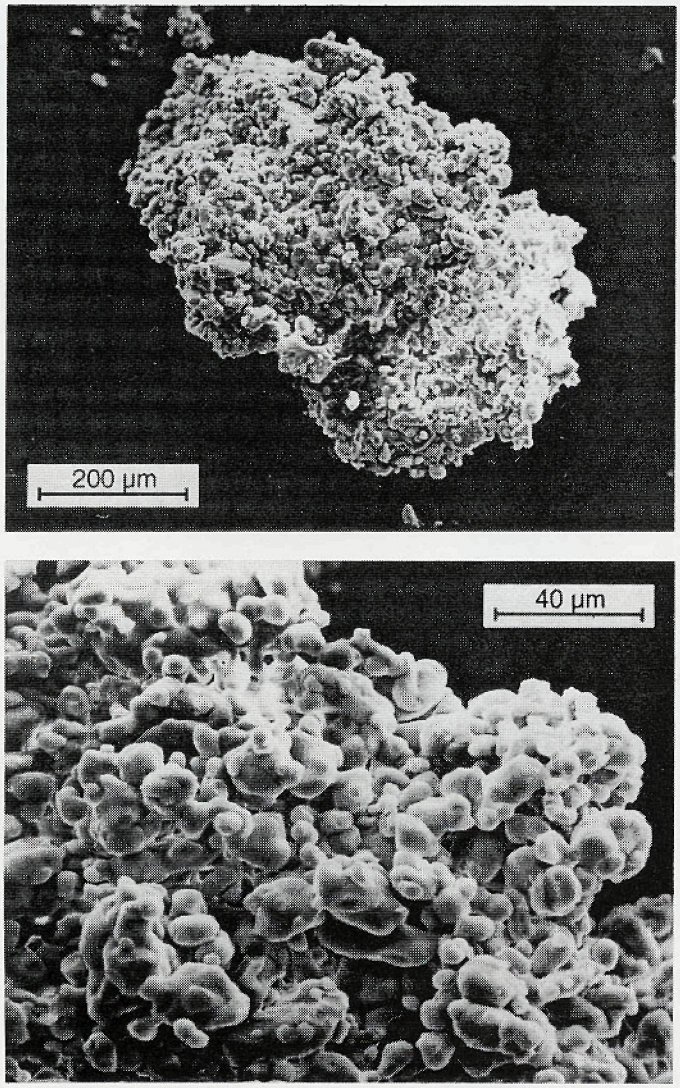

Figure 3. Two mesh fractions of undried AR grade potassium nitrate produced by ball milling for 12 hours. Upper, a particle in the range of 60 to 100 mesh at 100x magnification, lower, the same particle at 500x magnification.

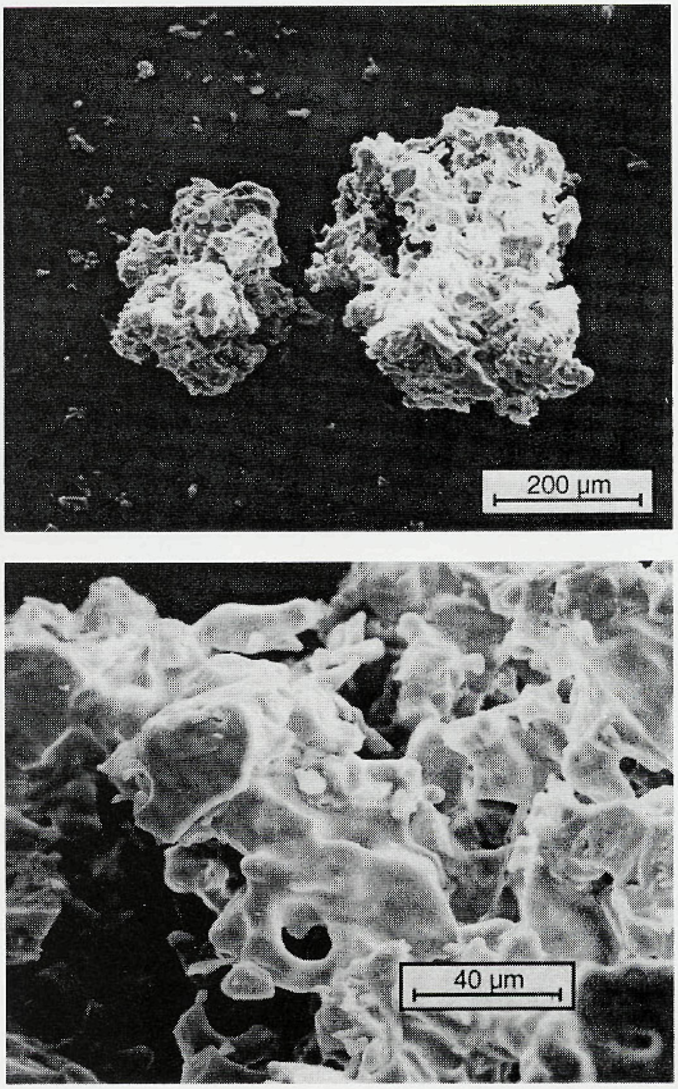

Figure 4. Particles from two mesh fractions of AR grade potassium nitrate produced by hot coacervation with rapid stirring. Upper, 60 to 100 mesh particles at 400x magnification; lower, a portion of the particle on the right at 500x magnification.

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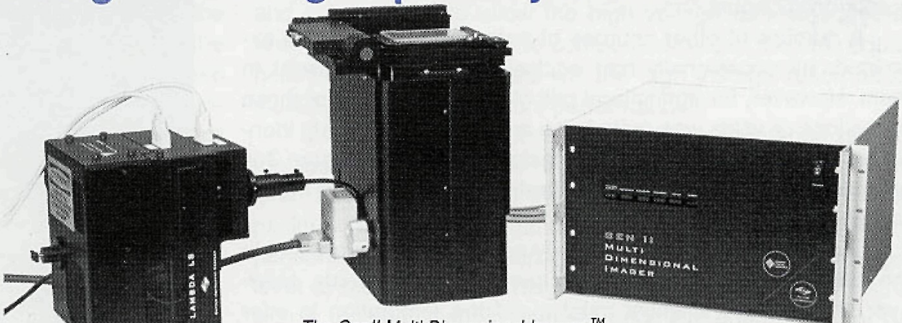

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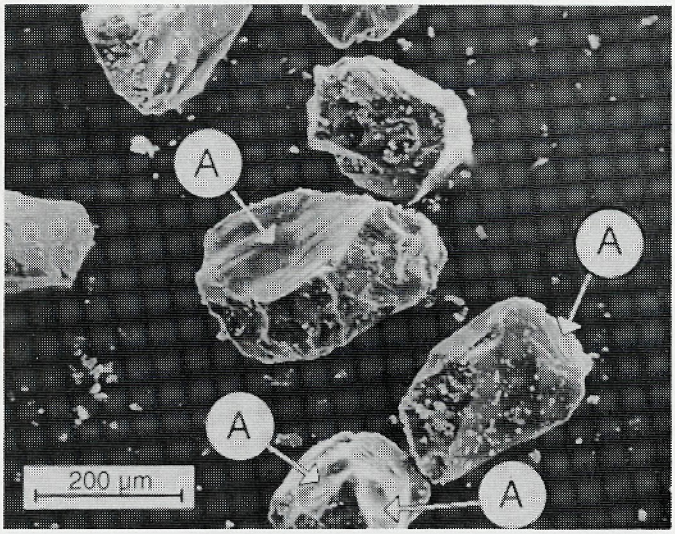
but for only 2 hours.

A number of other sources of potassium nitrate were examined; all occasionally had occlusions or voids present in them. However, for agricultural prill (AgP), the number of these occlusions or voids was quite large and can serve to help identify the source or be used to help establish a match. Figure 7 is an example of AgP potassium nitrate crushed using a mortar and pestle.

The use of particle morphology can be an important tool in the identification and matching of pyrotechnic materials. However, the forensic scientist must avoid the temptation to infer matches between samples and evidence based solely on particle morphology. There are other important characteristics that should be considered, such as trace chemical composition. There are also differences in crystalline structure that can produce different morphologies for the same chemical processed using the same method. ■



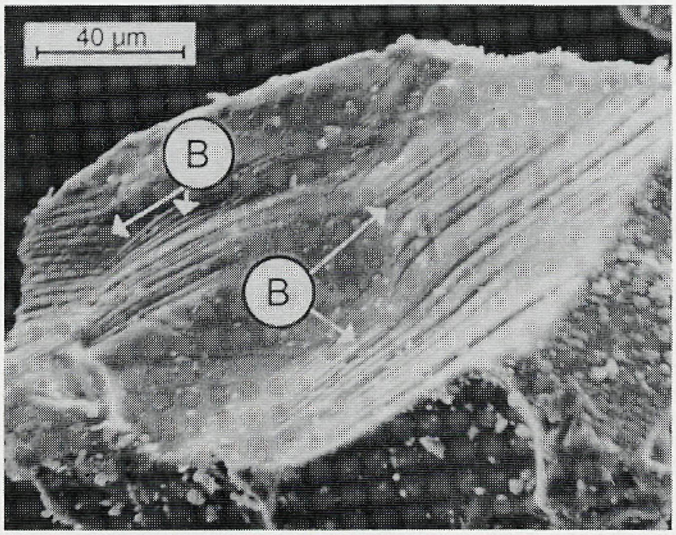
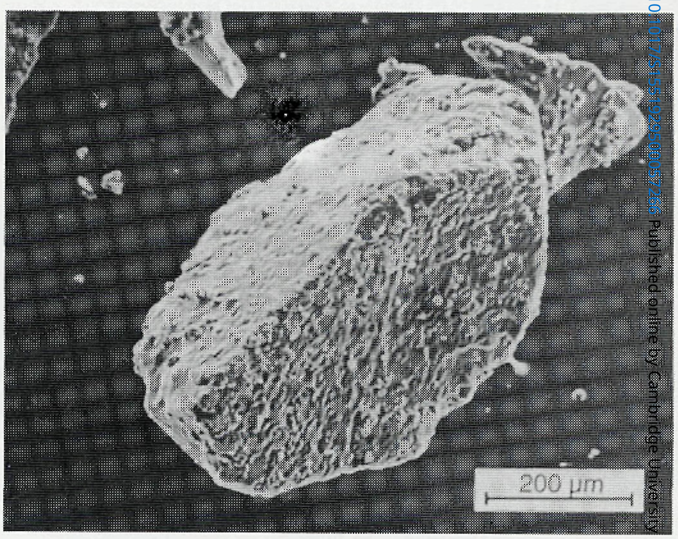


Figure 5. Particles of AR grade potassium nitrate produced by hammer milling, demonstrating fracture parerns (A) and a rather extreme number of "whiskers" (B). Upper, 100x magnification, lower, 500x magnification.



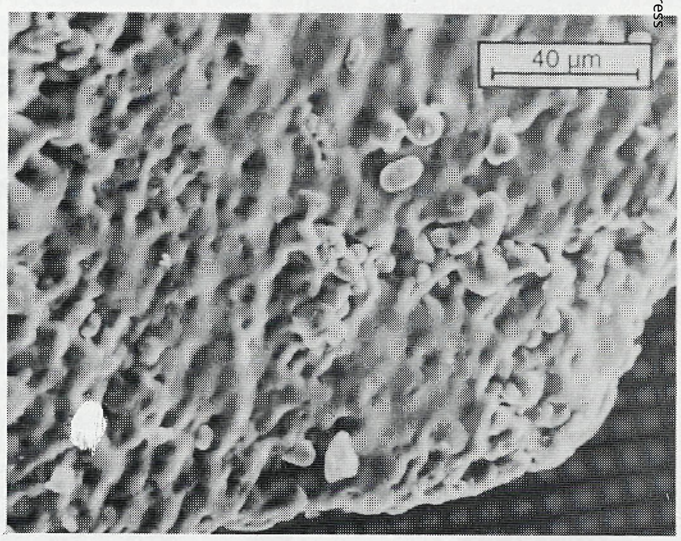


Figure 6. Particle of AR grade potassium nitrate produced by bal milling for 2 hours, illustrating the pitted surface of the 60 to 100 mesh particles, Upper, 100x magnification, lower, 500x magnification.

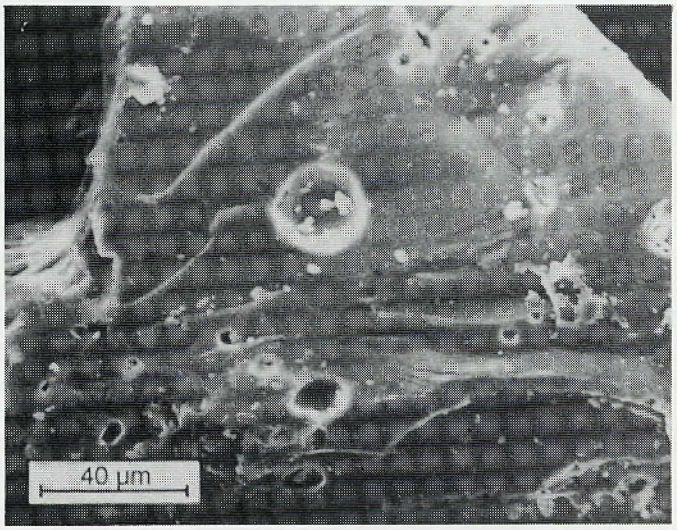


Figure 7. Particle of AgP grade potassium nitrate produced using a mortar and pestle, illustrating the presence of numerous occlusions, 500x magnification.

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