Analysis Of Unusual Home Made Shot Pellets From Louisiana

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CASE HISTORY

Thousands of migratory waterfowl over winter in the marshes, rice fields, and bayous of Louisiana. Although they may be hunted under Federal and State laws, birds are taken illegally every year. One method of illegally harvesting birds is via drive-by shooting. A typical drive-by shooting occurs on a road paralleling a shallow marsh or field where large numbers of waterfowl have congregated. The hunter will fire a shotgun into the thickest and closest group of birds while standing near or sitting in his vehicle. The hunter will then drive to a safe vantage point and wait to see if anyone responds to the sound of gun fire. If no one appears, the hunter will return to the scene and pick up the most accessible kills. Scores of birds die or are fatally crippled during these illegal shootings.

On January 15, 1992 a U.S. Fish & Wildlife Service Special Agent was patrolling a rice field area in Jeff Davis parish, Louisiana. At approximately 11:00 AM he heard five shots in rapid, even succession and saw a large flock of geese taking flight about 1/2 mile southeast of his location. He arrived at the scene within minutes. In a rice field adjacent to the road he observed fifteen to twenty dead or seriously crippled snow and blue geese. He also recovered two empty 12 gauge shot shells from the road. The shells had a "heavy fresh gunpowder smell to them". Based upon the location and availability of access to the rice field, he reasoned that the shooter was still in the area.

Two houses were located within 120 yards of the shooting scene. A suspect was interviewed at one of these residences. Two empty shot shells from the suspect's pick-up truck were voluntarily relinquished during the interview. Although the suspect allowed the special agent to examine his 12 gauge shotgun, which appeared to have been recently fired, he did not allow the agent to take it. After the interview, the special agent returned to the scene for documentation and to recover bird carcasses for evidence.

A search warrant was executed at the suspect's residence nine days after the shooting incident. Several loaded and spent shot shells and reloading equipment were confiscated during the search. The shotgun that the special agent had examined during his interview with the suspect was not found. During the execution of the search warrant a five gallon metal water bucket containing what appeared to be small gray metal pellets was observed.

The carcasses of one snow goose and one blue goose from the shooting scene were sent to the National Fish & Wildlife (NFW) Forensic Laboratory for necropsy. Radiographic examinations and necropsies of the geese revealed that they had been mortally wounded by shot gun pellets. Macroscopic examination and x-ray fluorescence spectrography of pellets removed from fresh wound tracks in the geese revealed the presence of lead shot in the 4/5/6/7½ size range. Also recovered was one heavily encrusted BB copper coated lead pellet which had been retained by one of the birds from an earlier, non-lethal wounding incident.

The expended shot shells from the scene and the live and expended shot shells from the suspect's truck and residence were sent to the Acadiana Criminalistics Laboratory for firearms comparisons. The Acadiana Criminalistics Laboratory's comparison of the scene shells to the suspect's spent shells showed that they were reloads which had been discharged by the same firearm. The live rounds were also determined to be reloads. Since the use of home made shot was strongly suspected in this case, pellets from two of the suspect's live rounds were forwarded to the NFW Forensic Laboratory for comparison against the pellets from the geese.

Scanning electron microscopic examination of the suspect's pellets to the only questioned lead pellet that survived with minimal impact damage revealed that they were consistent in unusual external morphology: atypical shapes, a broad size range and the presence of a single dimple like defect on each of the pellets. SEM/EDX also identified lead and antimony in both standard and questioned pellets. The morphological observations were subsequently shared with and confirmed by the Acadiana Criminalistis Laboratory.

On January 21, 1993 in a Lake Charles, Louisiana Federal Magistrate Court, following testimony by the investigator and State and Federal forensic scientists, the suspect was convicted of shooting geese from a public road, wanton waste of migratory birds, overlimit of geese and taking geese while in possession of lead shot. Part of the sentencing agreement required the suspect to cooperate with Federal officials in providing information and research samples in connection with his pellet manufacture activities. Photographs, a video tape and additional pellet samples were subsequently obtained and forwarded to the NFW Forensic Laboratory for examination.

METHOD OF SHOT MANUFACTURE

During post conviction verbal and video taped interviews, the defendant claimed that he melted down a variety of conveniently obtained objects such as sinkers, tire weights and lead pipe to make pellets. His mold was a large home-made long handled "aluminum" (metallic composition not verified) ladle (Fig.1). Lead was melted in a pot and poured into the mold. Perforations "about the size of a pin head" were drilled into the mold. The molten lead dripped through the holes into a bucket. The bucket contained water and oil (proportions not specified). The oil was selected on the basis of availability. Both vegetable oil and motor oil were listed. The estimated maximum free fall for the pellets between ladle and bucket was approximately four inches. The pellets were removed from the bucket and roughly sized using a "clover screen". Clover screens are agricultural devices used to separate seeds of differing sizes. The sized pellets were then loaded into shot shells.

ANALYSIS OF SHOT

Pellets were examined macroscopically, by low power optical microscopy, scanning electron microscopy/energy dispersive x-ray spectroscopy (SEM/EDX), x-ray fluorescence spectroscopy (XRF) and inductively coupled plasma atomic emission spectroscopy (ICP). Primary external examinations of both questioned and known pellets macroscopically and by SEM/EDX and qualitative elemental analysis by XRF were concurrent with the other State and Federal forensic examinations. Additional SEM/EDX and XRF examinations, optical examinations, and quantitative elemental analysis by ICP of additional suspect pellets were performed in preparation for this paper.

Photography and Optical Microscopy

Laboratory photographs were taken using a Cannon AE1 with a macro lense using 35 mm ISO 100 Kodak Kodacolor film. Optical examinations were performed using a Reichert Universal Forensic Microscope IV.

Scanning Electron Microscopy

Pellets were examined washed and unwashed in preparation for scanning electron microscopy and elemental analysis. Washed pellets were prepared by submersion in water and Fisher brand FL70 with gentle agitation. Pellets were secured to ¾" pin mount carbon stubs with STR brand double sided carbon tape. A Camscan Series 4 SEM with a Kevex 8000 energy dispersive x-ray spectrometer was operated at 25 kilovolts and 100 microamps for qualitative elemental analysis and 20 kilovolts with 100 microamps for imaging. Dead time for microanalysis was approximately 25% to 35% with a count rate of approximately 2000 counts per second. The acquisition time was 300 seconds. Micrographs were recorded on Polaroid type 54 film and Kodak 4162 negatives.

X-Ray Fluorescence Spectroscopy

Pellets were also examined using a Baird (formerly Asoma) EX-6000 x-ray fluorescence spectrometer. Samples were examined washed and unwashed in 1 inch sample cups with a polypropylene film base. The XRF rhodium x-ray source was set at 40 kilovolts and 25 microamps. Dead time was approximately 30% to 40% with a count rate of approximately 5000 counts per second. Acquisition time was 200 seconds.

Inductively Coupled Plasma Atomic Emission Spectroscopy

Quantitative elemental analysis was performed off site by IMO Baird Corporation of Boston, MA, using a Baird ICP 2000 one meter vacuum simultaneous inductively coupled plasma atomic emission spectrometer (ICP). Sample preparation for ICP consisted of dissolving 0.529 grams of shot in 10 milliliters of 1:1 nitric acid with gentle heating and then diluting the solution to 50 milliliters. A white crystalline precipitate that was observed after the sample had disappeared was dissolved by adding DI water and reheating the sample. The ICP was operated with the RF (radio frequency) generator at 1100 watts, coolant flow at 10.5 liters/min and auxiliary flow at 1.0 liter/min, nebulizer pressure at 43 psi, and sample flow 2 ml/min. The data collection was set for three integrations of five seconds.

RESULTS AND DISCUSSION

Macroscopic examinations of the two submitted suspect's shot loads revealed that the pellets were irregularly shaped and sized and each had a deep central dimple like depression defect which was visually apparent in many pellets (Fig. 2). The pellet sizes ranged from approximately 3.3 to 2.4 millimeters (4/5/6/7½ size range) in general diameter. The shapes of these pellets ranged from oval to tear drop with a number of intermediate variations. Perfectly spherical pellets such as the commercially produced Federal #6 lead pellets in Figure 3 were not observed. The pellets also appeared to be covered with a fine grit. Washing appeared to remove most of the "grit" from the pellets.

Examination of the relatively intact pellet removed from one of the bird carcasses revealed that it was rounded-oval, approximately three millimeters in diameter and contained a deep central dimple defect. SEM of this pellet (Fig. 4) revealed that the dimple defect was approximately 300 micrometers in width (Fig 5). The outer sides of the dimple depression were smooth. The dimple defects on the suspect's pellets (Figs. 6,7) ranged from approximately 100 micrometers to 1000 micrometers in diameter with smooth edges. Figure 8 is a scanning electron micrograph of Winchester #6 shot showing the typically spherical shape of commercially produced lead pellets.

Examination of the pellets suggests three possible (but not all inclusive) explanations for the presence of the dimple defect: shrinkage void, gas porosity, or an oxide or other inclusion [1,2]. Molten metal contracts as it "freezes" (solidifies). Solidification is generally progressive. A shrinkage void may form at the last area of solidification. Gas porosity results when gas that was trapped in the molten metal accumulates at the liquid-solid interface, which is also the last place to freeze. An oxide or other material may have been initially present on the pellet but was subsequently lost, leaving behind a hole or surface disruption. While the exact ontogeny of the dimple defect may remain unknown, the cause of this defect is probably related to the unusual manufacturing method employed by the suspect. Although shallow irregular surface pitts may occasionally be seen on commercially manufactured shot pellets, deep central dimple defects have not been observed on commercial shot to date.

Cross sectioning with a razor blade of three of the dimpled pellet standards also revealed the presence of an approximately 250 to 600 micrometer cavity beneath the dimple defect (Fig.9). A direct connection between the cavity and the dimple defect was not observed. The cavities displayed dendritic side walls (Fig.10). It is uncertain whether this cavity is directly associated with the process that produced the dimple defect or if it formed independently. The questioned pellet was not sectioned.

The formation the dimple defect may be batch related. Shot from the two suspect reload shells originally received as case comparison standards displayed the dimple defect. Shot from twelve other suspect reloaded shells have since been examined. One (Remington low brass duplex 7½ X 8) shell contained commercially produced #2 buckshot and one (Remington high brass #7½) shell contained what may have been severed halves of small (approximately .15" diameter) fishing line weights. Ten shells contained pellets in the size and shape range of the previously examined suspect pellet standards. Among these ten shells, one load consisted of dimpled pellets, one contained pellets with small central knobs, and eight loads displayed few if any significant surface anomalies by low power optical microscopy. SEM examination of these pellets revealed shallow surface disruptions.

Qualitative elemental analysis of the questioned pellet and dimpled and non-dimpled pellet standards by SEM/EDX and XRF revealed the presence of lead and antimony. Both of these elements are frequently detected in commercially produced shot.

Quantitative elemental analysis of dimpled standard pellets by ICP revealed the presence of 96.38% lead, 1.29 % antimony,

0.04% tin, 0.02% arsenic and 0.01% bismuth with several other metals showing trace amounts equal to 0.007% or less. Aluminum registered less than 0.0001% and iron .0004%. This analysis reflects the combined composition of approximately five pellets. Batch to batch variability would not be unexpected considering the variety of lead based objects the suspect alleged to have used in his shot making operation.

Previously published quantitative elemental analysis of commercially produced lead shot reported 0.5% to 6.5% antimony, and 0.1% to 1.0% arsenic [3,4,5]. Tin may also be present in the 0.1% range [6]. Bismuth, possibly present as a lead ore impurity, has been detected in trace amounts in bullet lead [7].

CONCLUSION

The exceptional external morphology of home made shot pellets in this case was observed and presented as circumstantial evidence of association between an illegal act and an individual suspect.

Routine qualitative elemental analysis of the questioned and standard pellets revealed the presence of lead and antimony. Subsequent quantitative analysis of suspect standard pellets by ICP revealed that the amounts of these elements were within the expected ranges for commercially manufactured shot although the trace elemental components differed from norm.

ACKNOWLEDGEMENTS

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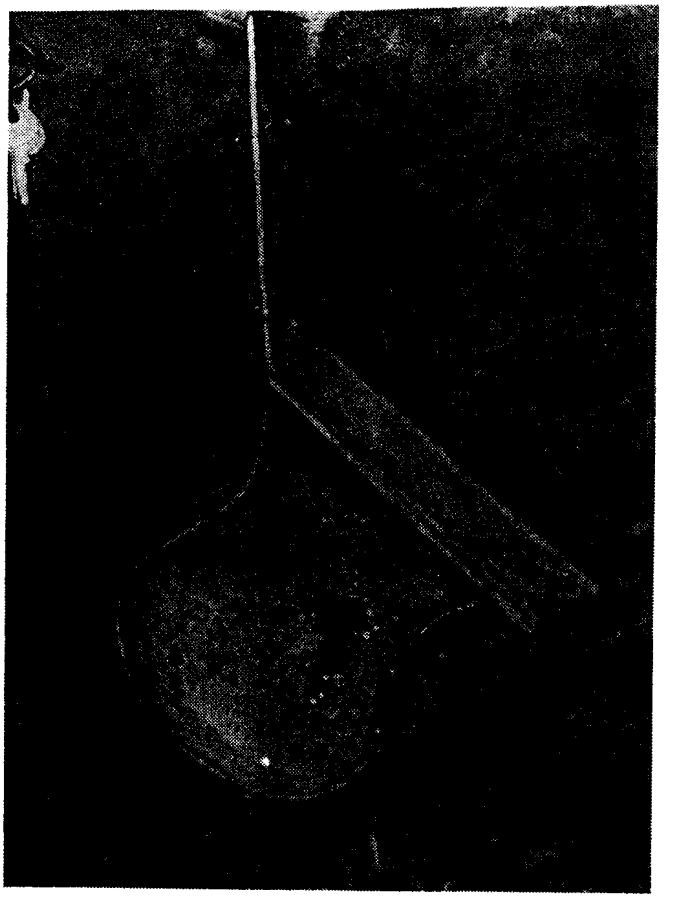

Fig. 1 Photograph of the "mold"/ladle used by the suspect to make lead shot.

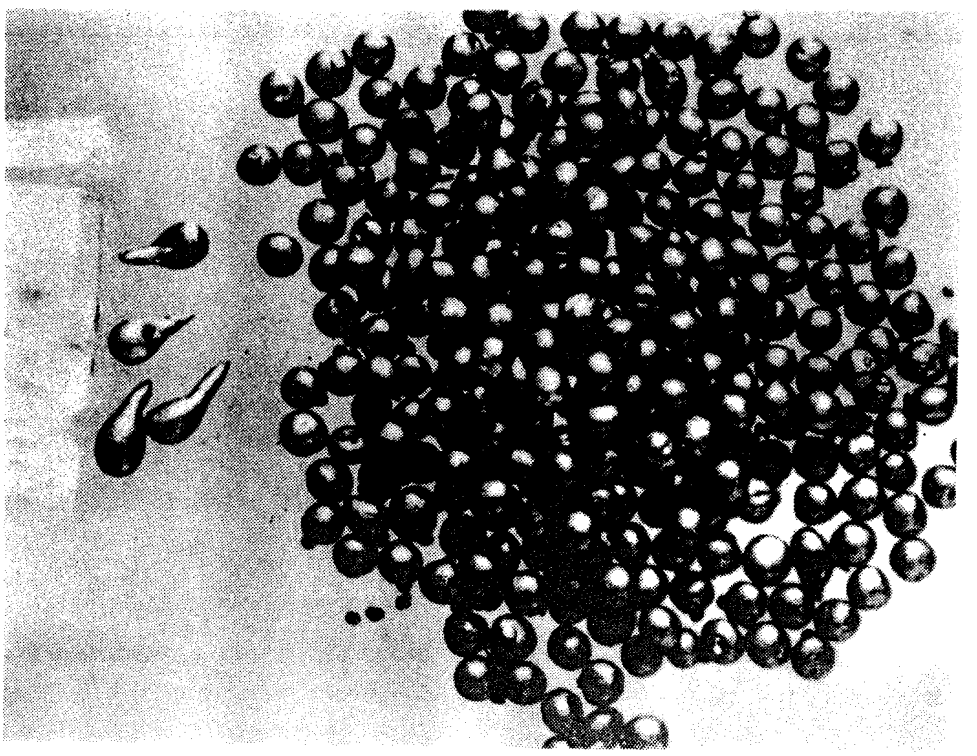

Fig. 2 Macroscopic image of one of the suspect's loads. Notice the variety of shapes and sizes. The dimple defect is visible on several of the pellets.

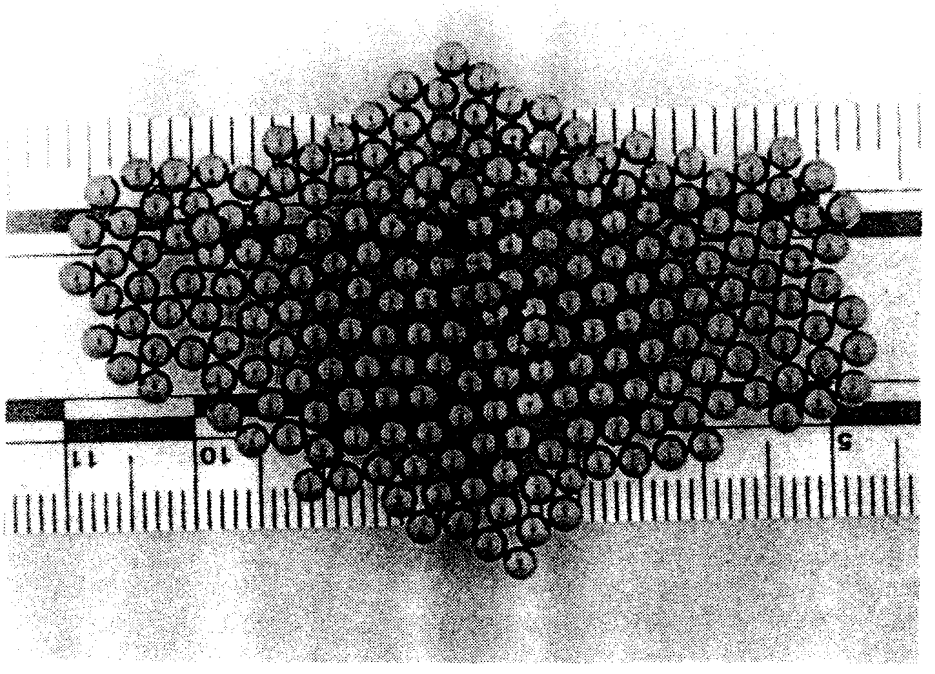

Fig. 3 Macroscopic image of commercially produced Federal #6 shot pellets.

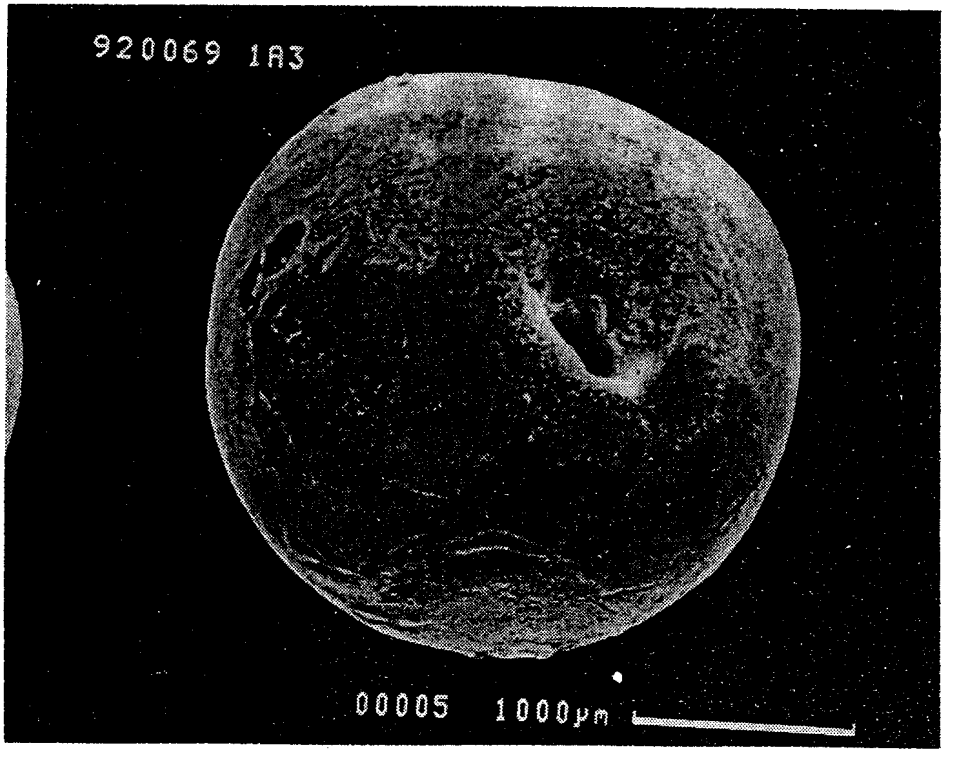

Fig. 4 Scanning electron micrograph of an intact questioned pellet removed from a goose carcass. Original magnification X30.

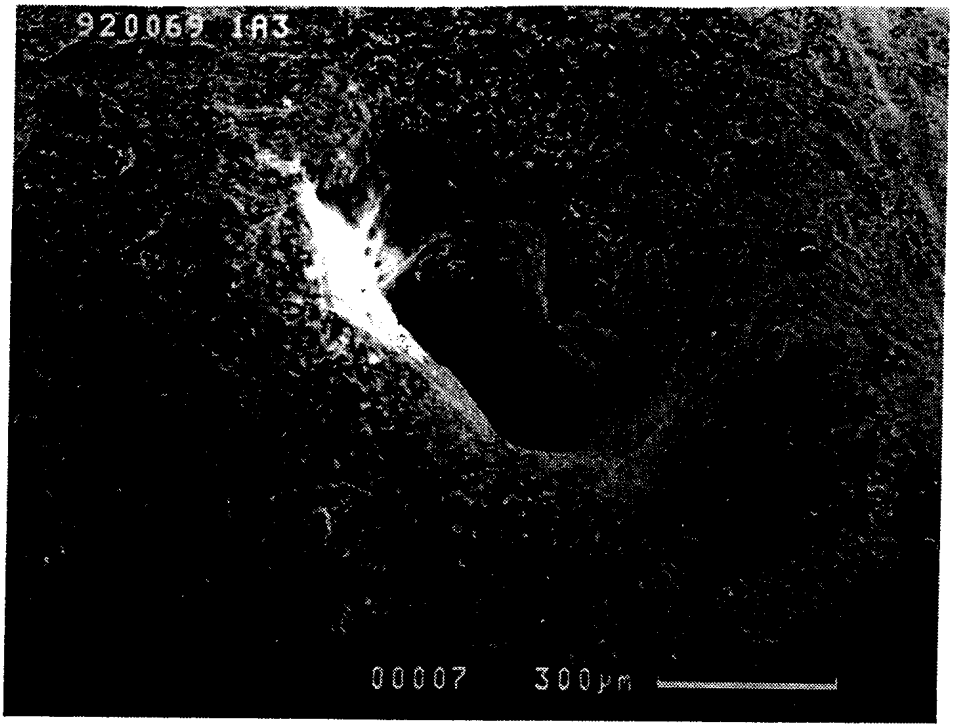

Fig. 5 Scanning electron micrograph of the dimple defect in the questioned pellet shown in Fig. 4. Original magnification X70.

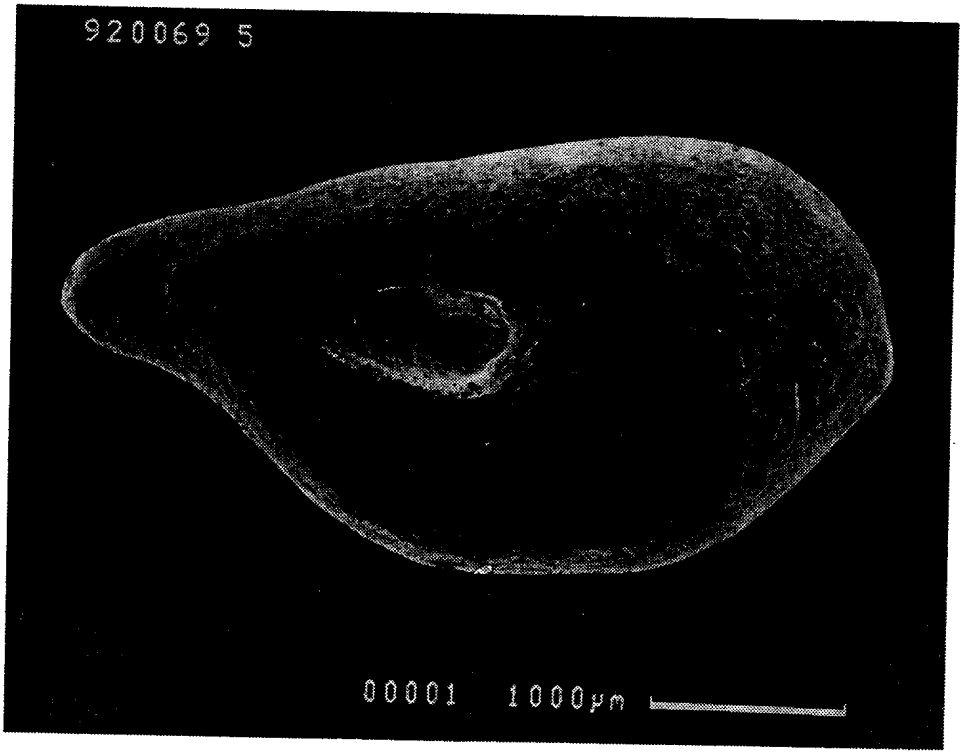

Fig. 6 Scanning electron micrograph of one of the suspect's pellets showing the dimple defect. This pellet is tear drop shaped. Other pellet shapes included oval and rounded. None of the suspect's pellets were perfectly spherical. This pellet was examined unwashed. Original magnification X25.

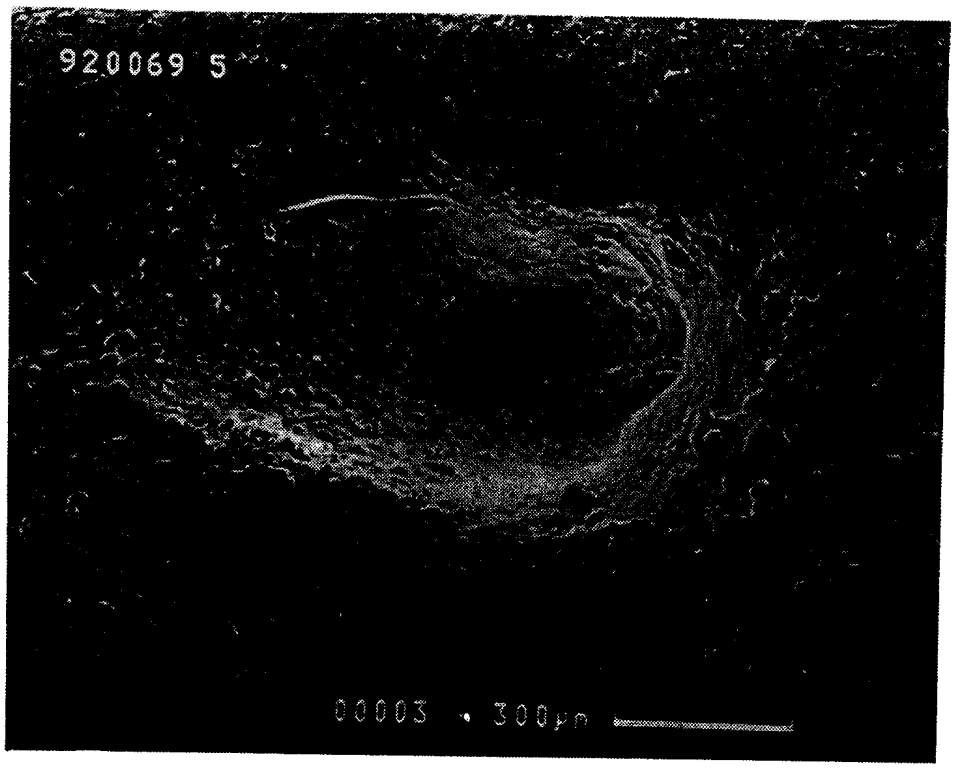

Fig. 7 Scanning electron micrograph of the dimple defect in the suspect's pellet shown in Fig. 6. Original magnification X70.

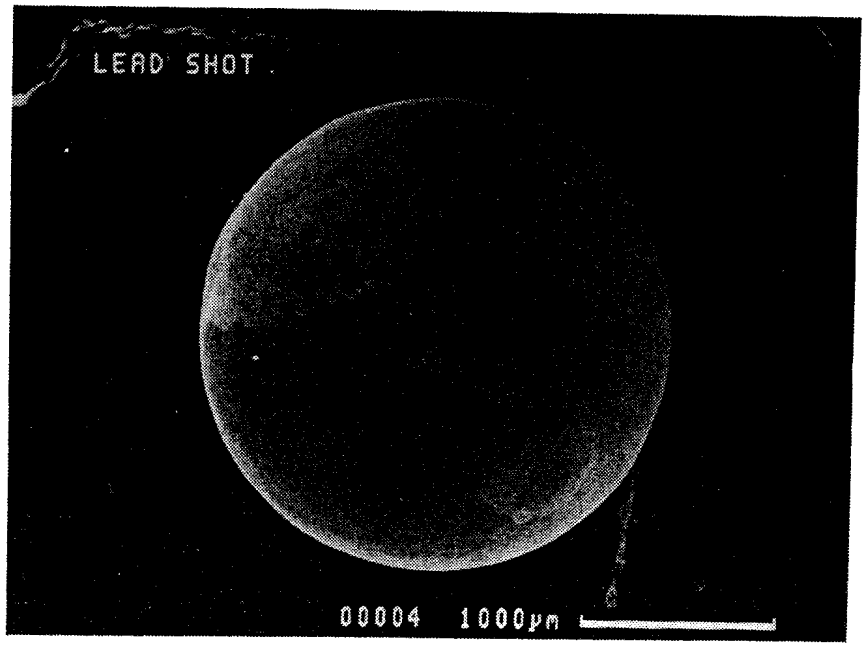

Fig. 8 Scanning electron micrograph of a commercially produced Winchester #6 shot pellet. Original magnification X25.

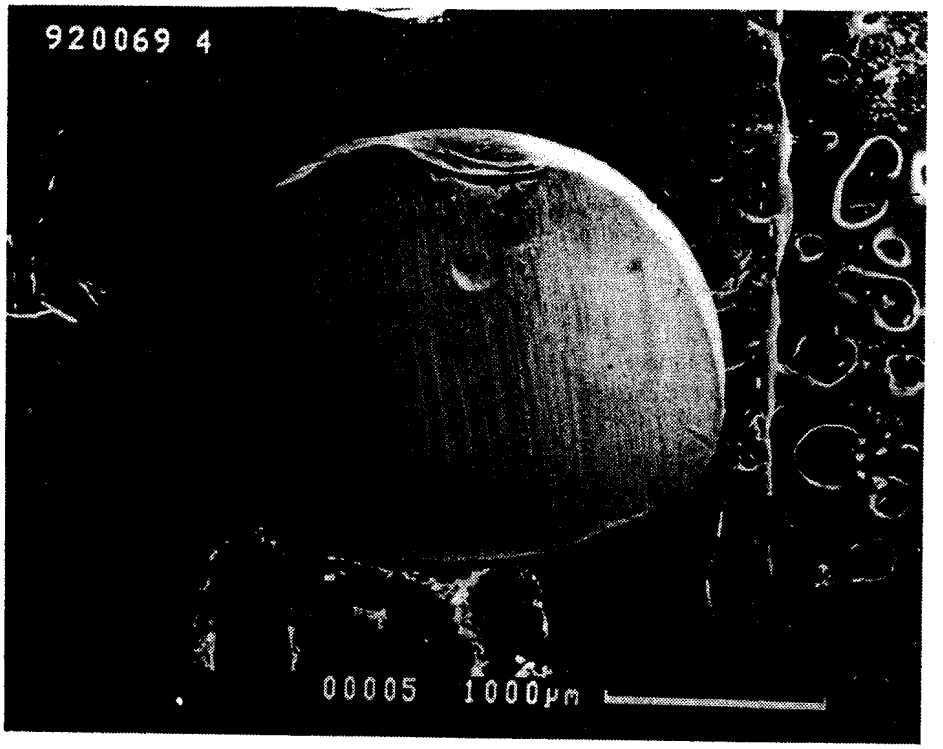

Fig. 9 Scanning electron micrograph of a razor blade cross-section of one of the suspect's pellets showing a cavity beneath a dimple defect. It is not known if the cavity and the dimple defect are associated phenomena. Original magnification X30.

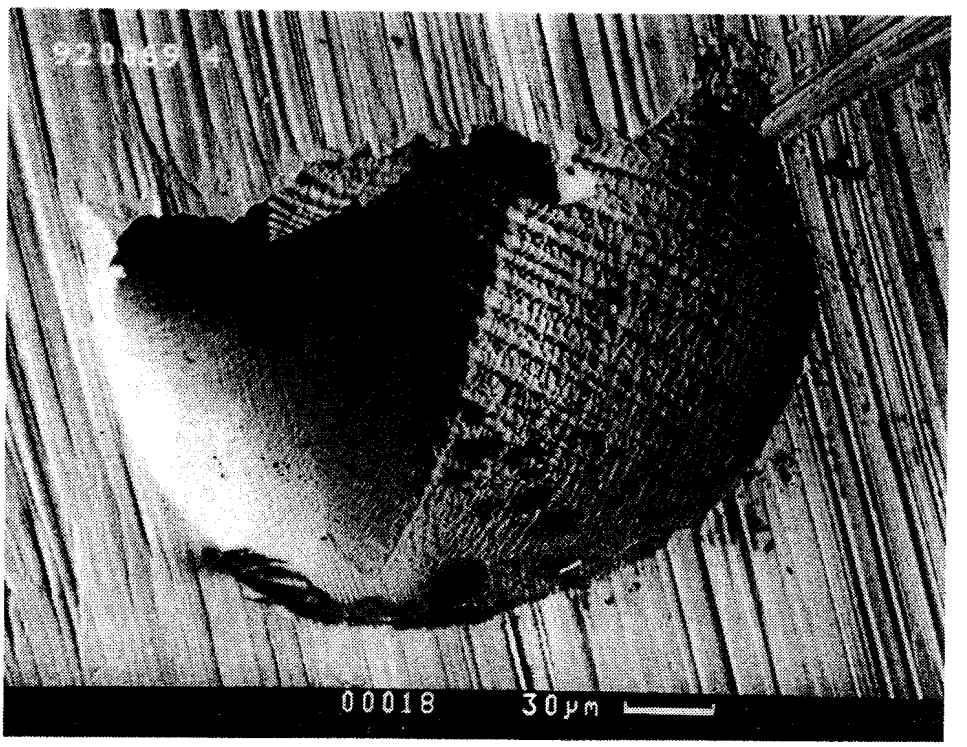

Fig. 10 Scanning electron micrograph of the cavity seen in Fig. 9. Notice the dendritic side walls. Original magnification X350.