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Technical Report ARMET-TR-11001

ALUMINUM-ZINC (Al-Zn) DUST ANALYSES FOR RECYCLING PURPOSES

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14. ABSTRACT

The physical vapor deposition (PVD) process facility at the McAlester Army Ammunition Plant, McAlester, Oklahoma coat 500-lb bombs with aluminum-zinc (Al-Zn). Transmission electron microscopy and electron diffraction technique revealed Zn and Al particles in the dust waste generated by this PVD process. The x-ray fluorescence analyses and selected electron diffraction patter confirmed the presence of Al-Zn particles and their crystal structure. Powder data files and a standard evaporated gold thin film electron diffraction pattern were used to determine the d-spacing of Al and Zn dust particles.

15. SUBJECT TERMS

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INTRODUCTION

The U.S. Army Armament Research, Development and Engineering Center (ARDEC), Picatinny Arsenal, New Jersey engineers visited the McAlester Army Ammunition Plant (MCAAP), McAlester, Oklahoma on 10 July 2008 to view and obtain information on their zinc (Zn) and aluminum (Al) disposal process. For the past 2 yrs, the price to dispose of this waste has increased significantly. The original cost to remove the Al/Zn dust was set at $0.39/lb (FY08). In 1 yr, the price increased to $2.00.lb (FY09). In the past year, this price escalated to approximately $6.00/lb (FY10). This translates to an estimated cost of $400,000 a year to dispose of this waste. The main reason for this spike in price is due to the reactivity of the collection drums. The Al in the dust particles reacts with rain water. This reaction results in hydrogen gas, which builds up pressure. The barrels are occasionally mishandled and sealed with water inside. The buildup of pressure results in the barrel's top firing off. This increased hazard increased the shipping cost dramatically. Unless an alternate form of disposal is found for this Al-Zn waste, the physical vapor deposition (PVD) process facility will be closed. The primary purpose of the PVD thermal arc process metal coating is to provide fire protection for the round and increase the bomb's high temperature threshold. In addition, it provides a more effective adhesion of paint to the shell body.

The ARDEC engineers contacted the Sarakem Corporation, a trusted hazardous waste management industry in Hanover, New Hampshire. The composition of Al-Zn dust and total weight of shipment was determined and provided to the Sarakem Corporation that planned to recycle this dust to zinc oxide as a chemical product. The Al-Zn waste stream that was previously disposed of as hazardous waste was reclassified as a recyclable waste, since MCAAP/ARDEC secured a contractor (Sarakem) that will accept and recycle the Al-Zn waste stream. This resulted in a total cost savings of approximately $838,860.

SPECIMEN PREPARATION

The 500-lb bombs are coated with Al-Zn (fig. 1) and the Al-Zn dust is collected in 50-gal drums (fig. 2). The ARDEC received this Al-Zn dust from MCCAP for x-ray fluorescence (XRF) analysis to determine the composition of the dust sample.

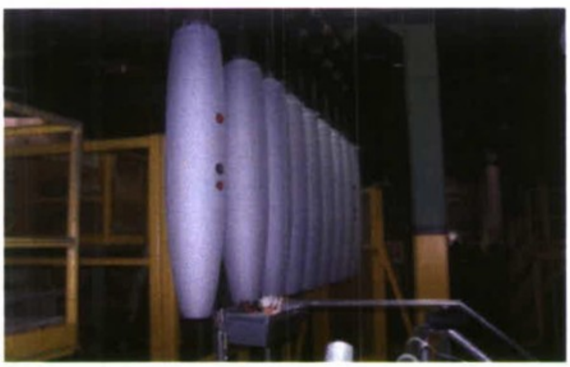

Figure 1
500-lb bombs with Zn-Al coat

Figure 2
50-gal drums stored at MCAAP

A small amount of this Al-Zn dust was dissolved in water and dropped on a 200-mesh carbon coated copper grid and dried at room temperature. This specimen was then placed in a single tilt specimen holder and inserted in a Philips 420 transmission electron microscope (TEM).

DISCUSSION/CONCLUSIONS

The XRF analysis revealed approximately 88.8% Zn, 10.9% Al, and minute traces of other metal elements as shown in the table.

SQX Calculation Result

Sample :
Application : EZS003MSV Model : Bulk
Date analyzed : 2009- 2-27 15:49
Balance :
Matching library:
File : Tapan_AlZn

No.	Component	Result	Unit	Det.limit	El.line	Intensity	w/o normal
1	Al	10.9	mass%	0.0243	Al-KA	15.3782	10.3946
2	Si	0.143	mass%	0.0060	Si-KA	0.1998	0.1358
3	P	0.0053	mass%	0.0027	P -KA	0.0218	0.0050
4	S	0.0167	mass%	0.0030	S -KA	0.0567	0.0159
5	Ca	0.0237	mass%	0.0104	Ca-KA	0.0949	0.0225
6	Fe	0.0877	mass%	0.0078	Fe-KA	0.7050	0.0833
7	Ni	0.0129	mass%	0.0052	Ni-KA	0.1920	0.0122
8	Zn	88.8	mass%	0.0236	Zn-KA	889.4754	84.3667

Transmission Electron Micrographs Analyses

The TEM of Al-Zn dust samples were taken from different specimens at low and high magnifications. Figure 3 shows a Zn particle denoted by A; the arrow indicates the border of a single mesh. The same area was blown up to a higher magnification and figure 4 shows an area with a large a concentration of Zn particles and a much smaller concentration of Al dust particles are clustered together. Figure 5 shows an electron micrograph of very high magnification from another area of the specimen. Clusters of Al-Zn dust are revealed. A selected electron diffraction (SAED) pattern corresponding to this electron micrograph is shown at figure 6. The spotty rings reveal the Al-Zn dust particle distribution is not polycrystalline in nature; rather they are preferentially oriented. Low electron beam density and a small condenser aperture were used to obtain this SAED. Figure 7 is another SAED pattern taken from a different area of the specimen. A larger condenser aperture with normal electron beam density was used to obtain this ring pattern that is different from that in figure 6.

It is concluded the Al-Zn dust particles generated by the PVD deposition process are not homo-geneous in nature. The d-spacing's of these ring patterns were measured using the equation $L\lambda=rd$; where L= camera length, λ is the wavelength of the electron beam at 120kV, r = radius of the diffraction ring, and D is the interplanar spacings of the Zn and/or Al particles. The powder data file and a standard electron diffraction pattern (fig. 8), from standard Au specimen, confirmed the presence of Al-Zn particles.

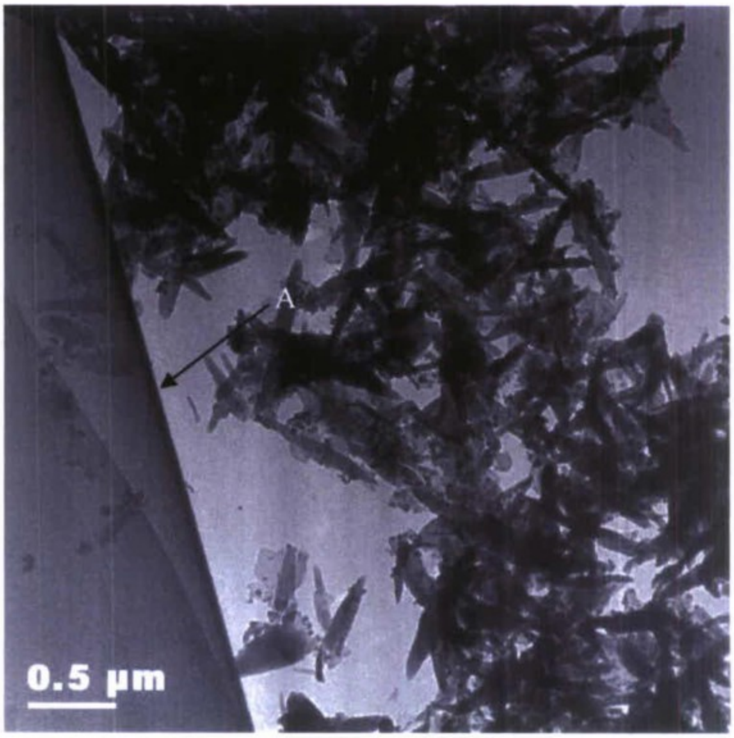

Figure 3
TEM of Al-Zn dust on 400-mesh grids

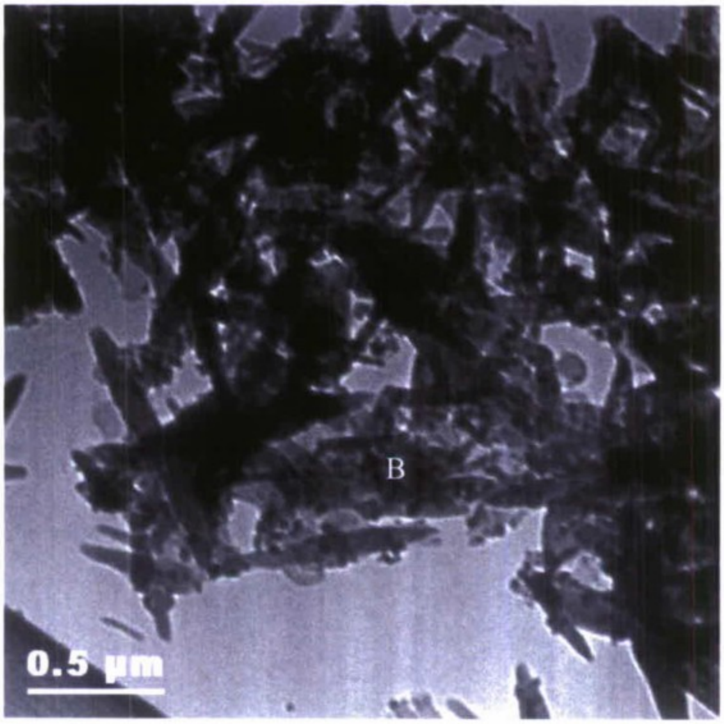

Figure 4
Magnification of this TEM picture is higher than that of the figure 3

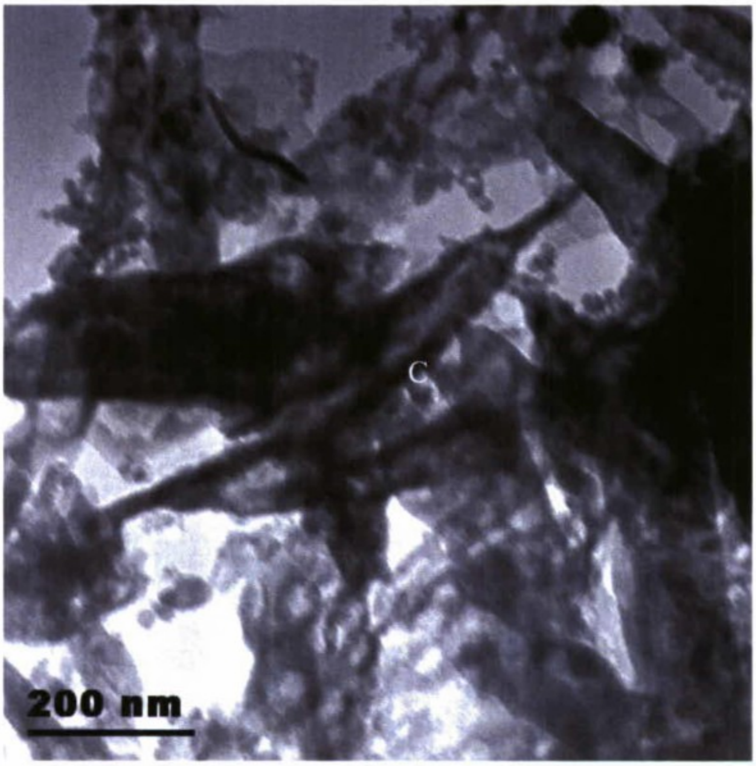

Figure 5
400-mesh grid at very high magnification at nano range
(different area)

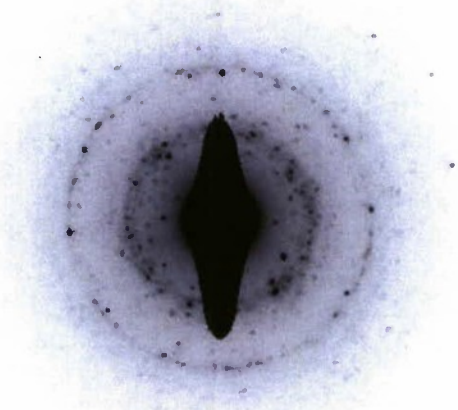

Figure 6
Electron diffraction pattern from Al-Zn dust with very low beam density

Figure 7
Electron diffraction pattern of Al-Zn dust with normal beam density using a beam stop

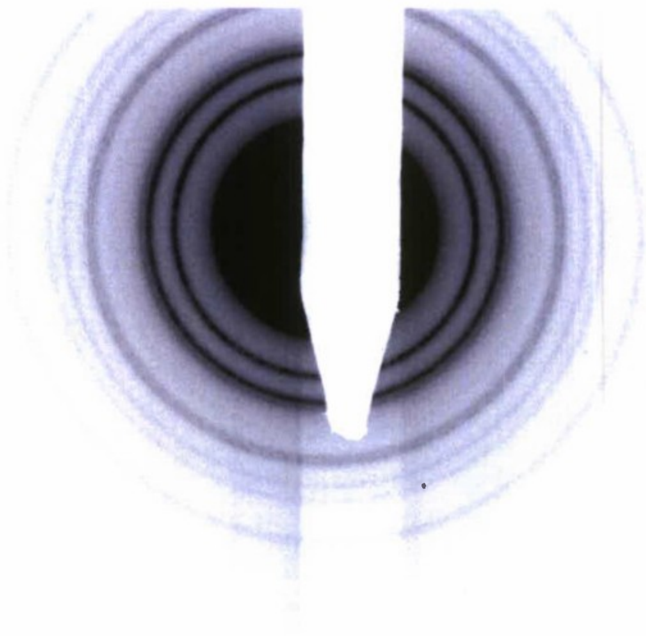

Figure 8
Electron diffraction pattern from standard gold sample used for calibration

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