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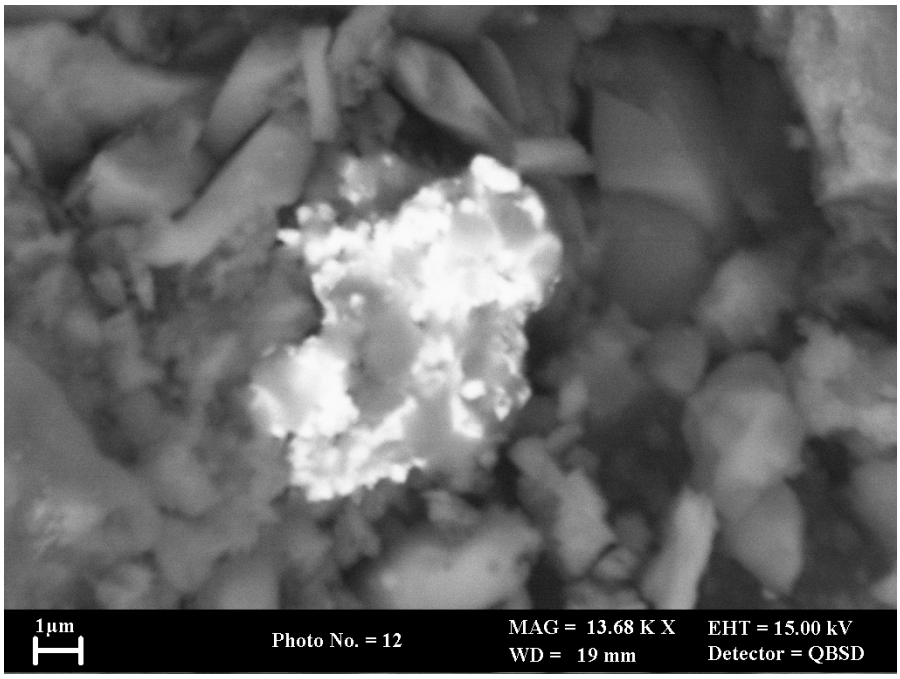
A trial investigation by scanning electron microscopy of uranium particulate in building debris associated with a Depleted Uranium munitions strike site during the Kosovo Conflict

Pollution and Waste Management, and Extraction Industry Impacts Programme

Commercial Report CR/01/145N



BRITISH GEOLOGICAL SURVEY

COMMERCIAL REPORT CR/01/145N

A trial investigation by scanning electron microscopy of uranium particulate in building debris associated with a Depleted Uranium munitions strike site during the Kosovo Conflict

AE Milodowski

Key words

Depleted uranium, NATO, Kosovo, armour piercing, concrete, cement, munitions, scanning electron microscopy, SEM

Front cover

Cover picture: backscattered scanning electron microscope image of surface of a fragment of a roof tile from the VJ Barracks, Kosovo, showing composite particle of uranium rich material (bright) and aluminosilicate material (dull).

Bibliographical reference

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Foreword

This report has been produced by the British Geological Survey (BGS) on behalf of DERA as part of an investigation into the environmental and health risk associated with the contamination by depleted uranium (DU) used in armour-piercing munitions fired by NATO aircraft at Serbian targets in Kosovo. DERA/MoD undertook sampling of soils and building debris associated with DU munitions strike sites in Kosovo. Two samples of building debris were studied in detail by BGS, using scanning electron microscopy (SEM), to characterise the nature of potential DU contamination.

Contents

Summary

Recent public debate has highlighted the concern over the potential long-term environmental and health effects arising from the use of depleted uranium (DU) munitions in recent military conflicts (Royal Society, 2001). DU munitions were used by NATO anti-tank (A-10) aircraft against Serbian targets during the recent conflict in Kosovo. In the United Kingdom, DERA are investigating the radiological and toxicological risk from DU munitions used in Kosovo. On behalf of DERA, the British Geological Survey (BGS) undertook the analysis of two samples of building debris from a DU munitions strike site (VJ Barracks) to characterise the nature of uranium-rich particulate material in DU-contaminated material. The samples were analysed by scanning electron microscopy techniques, using backscattered scanning electron microscopy (BSEM) to locate the uraniferous particles, and secondary electron imaging (SEM) to observe the morphology of the particles. Phase identification was supported by semi-quantitative microchemical information obtained from the phases by simultaneous energy-dispersive X-ray microanalysis (EDXA) during BSEM and SEM observation. This report details the analytical methods used and presents the observations and conclusions from this study.

BSEM proved very successful in locating dense uranium particulates on the surfaces of masonry fragments (cement mortar, concrete dust and fired-clay roof tile) because the very high atomic number of uranium produces high electron backscattering effects. Uraniferous particles were found on masonry debris in both samples. It is concluded that this represents DU dust since no similar uraniferous material was found within freshly broken surfaces of the masonry material.

The uraniferous particulate material falls into two size classes: (i) very fine material with a particle size less than 2 μm and generally less than 0.5 μm; and (ii) coarser particles, typically 10-15 μm in size, which comprise aggregates of finer uraniferous material with calcium silicate or aluminosilicate material. None of the uraniferous particles observed illustrated the typical spherical morphology of DU particulate reported previously from test firing of DU munitions against hard targets (cf. Patrick & Cornette, 1978). Instead, the observed DU particles from the VJ Barracks debris are mainly flaky in morphology (single particles) or aggregates of flaky or diffuse material.

The DU particulate displays a variety of compositions. Some material is possibly partly oxidised microscopic uranium metal shards. This is particularly the case for particles found on the roof-tile fragments. However, particles found on cementitious debris (either cement mortar or concrete dust) are more complex, varying from uranium oxide to calcium-uranium silicate or calcium-uranium aluminosilicate phases. Although it is possible that the EDXA analyses obtained from very fine uraniferous particulate are influenced by background X-ray emission from the cement substrate, observation of coarser particles (>10 μm) suggest that in the calcium, silicon (and aluminium) are intrinsic to the uraniferous particle itself. This implies that there has been reaction between the uranium and calcium silicate or calcium aluminosilicate matrix of the cement. There is also evidence that some of the DU particulate has been altered to a uranium carbonate phase.

1 Introduction

Recent public debate has highlighted the concern over the potential long-term health effects arising from the use of depleted uranium (DU) munitions in recent military conflicts (Royal Society, 2001). DU munitions were used by Allied forces in the Gulf War in 1991 and more recently by NATO in Bosnia and Kosovo. Reports of subsequent illnesses (e.g. 'Gulf War Syndrome') suffered by some the combatants involved in these conflicts has drawn attention to the possibility that they may be related to exposure to DU.

Uranium is a very dense metal (about twice as dense as lead) which exists in nature as three isotopes, ^{238}U, ^{235}U and ^{234}U. ^{238}U and ^{235}U are the most abundant isotopes and represent primordial uranium. ^{234}U is the radioactive decay product of ^{238}U. All three isotopes are unstable and are radioactive. Uranium is also a toxic heavy metal. DU is a by-product of the nuclear fuel manufacturing process, whereby nuclear fuel is enriched in the more radioactive ^{235}U. As a consequence, the residual uranium, which is depleted in ^{234}U and ^{235}U (i.e. DU), is about 40% less radioactive than natural uranium (Royal Society, 2001). However, the chemical toxicity of DU is the same as natural uranium.

As a by-product of nuclear fuel manufacture, DU is both abundant and cheap. Consequently, it has found cost-effective use in radiation shielding, rotors, and flywheels and in counterbalances for aircraft control surfaces. Its very high density and metallurgical properties also make DU ideal for military use as a kinetic energy 'penetrator' in munitions rounds designed to penetrate heavy armour of modern battle tanks and other armoured vehicles. These rounds are non-explosive but rely on the very high kinetic energy of a very dense DU projectile, travelling at high speed, to 'punch' holes in sophisticated modern armour plating. Unlike alternative tungsten penetrators, which blunt on impact with heavy armour, penetrators made of DU (alloyed with a small amount of titanium) are 'self-sharpening'. Consequently, DU penetrators have considerably greater penetrative properties than alternative armour-piercing rounds. Furthermore, when a DU penetrator strikes tank armour it generates a dust cloud within the vehicle which spontaneously combusts, creating a fire which enhances damage to the target. DU can also be used in defensive armour plating of modern tanks, where it gives significantly increased protection against armour-piercing munitions.

There are concerns over the health risk posed by short-term exposure of military and civilian personnel to DU through handling the munitions during the conflict, or from inhalation or ingestion of the DU dust (created by impact of DU penetrators) under battle conditions or during post-conflict clean-up operations. Also, very little is known about the longer-term geochemical behaviour of DU contamination in the environment and the health risks that this may pose in the future. In the United Kingdom, DERA are investigating the radiological and toxicological risk from DU munitions used in Kosovo. On behalf of DERA, as part of this assessment, the British Geological Survey (BGS) has undertaken a small trial investigation to characterise the nature of uranium-rich particulate material in DU-contaminated material in building debris from the VJ Barracks in Kosovo – which was struck by DU munitions fired from NATA A-10 anti-tank aircraft. Two samples of building debris were examined in detail by scanning electron microscopy. The objectives were to:

- determine whether the presence of particulate uranium matter could be identified in the samples;
- identify the physical and chemical characteristics of uranium-rich particles in the samples;
- describe any evidence of alteration of uraniferous material in the samples.

2 Analytical method

2.1 SAMPLE DETAILS

Two samples of building rubble, collected from the vicinity of the VG Barracks building in Kosovo, were studied. Both samples were monitored for alpha and gamma radioactivity using a Nuclear Enterprises Ltd Alpha Probe AP 2/4 alpha detector and a MiniMonitor Series 900 with a 44A total dose detector (15-500 KeV range). A summary description of each sample is given below:

DERA Sample 61/001 (BGS SEM reference code G900)

> Composite sample of rubble comprising orange-red fired-clay tile fragments (presumed to have been derived from roof tiles) and buff-white cement mortar, some of which is attached to tile fragments, and is presumed to be the mortar from between roof tiles. The sample was taken adjacent to a DU penetrator impact site.
>
> Surface gamma activity = 12-15 counts per second (background = 10 counts per second).
> Surface alpha activity = 0 counts per second (background = 0 counts per second)

DERA Sample 61/003 (BGS SEM reference code G901)

> Fine cement dust and small concrete fragments (up to 3 mm diameter) sampled from concrete structure, and taken and extracted from a location immediately adjacent to a DU penetrator impact entry point.
>
> Surface gamma activity = 12-15 counts per second (background = 10 counts per second).
> Surface alpha activity = 35-40 counts per second (background = 0 counts per second)

2.2 SCANNING ELECTRON MICROSCOPY

2.2.1 Sample preparation

Samples for analysis by scanning electron microscopy (SEM) were prepared as SEM stub mounts. Two separate SEM stub mounts were prepared from DERA Sample 61/001: (a) fragments of cement mortar; and (b) fragments of fired-clay roof tile. Two duplicate SEM stub mounts were prepared from DERA Sample 61/003.

The SEM stub mounts of DERA Sample 61/001 were prepared by hand-picking and fixing small fragments (<1 cm^2) of tile or cement onto 13 mm diameter aluminium pin-type SEM stubs, using Leit CCC conducting carbon cement. Tile fragments were mounted so as their 'flat' (i.e. unbroken) surfaces were able to be examined in the SEM. The SEM stub mounts of DERA Sample 61/003 were prepared by carefully sprinkling cement dust and granular material onto the surface of a pin-type SEM stub coated in a veneer of Leit CCC conducting carbon cement.

Prior to examination in the SEM instrument the stub mounts were coated with a thin film (approximately 25nm thick) of carbon to make their surfaces electrically conductive. Carbon coating was carried out using an Emitech 950 vacuum evaporation-coating unit.

2.2.2 SEM examination

SEM analysis was carried out using a LEO 435VP variable pressure digital scanning electron microscope, fitted with an Oxford Instruments ISIS 300 digital energy-dispersive X-ray microanalysis system (EDXA) system and a KE-Developments four-element solid-state backscattered electron (BSEM) detector. Observations were typically made in conventional high vacuum SEM mode, using 10-20 kV electron beam accelerating potential and 50 to 500 pA

beam currents, as required to achieve the desired observational resolution. The samples were imaged using both secondary electron imaging (SEI) and backscattered electron imaging (BSEM). However, where sample charging problems were experienced (due to poor adhesion of the carbon coating) the instrument was operated in variable pressure mode (at 0.1-0.3 torr) using backscattered imaging only. Phase identification was aided by semi-quantitative energy-dispersive X-ray microanalysis of features of interest using the EDXA system simultaneously during SEM/BSEM observation.

The surfaces of the samples were initially scanned under BSEM to locate uranium-rich particles. The BSEM image brightness is proportional to the average atomic number of the material, thus allowing the distribution of different minerals or phases to be determined on the basis of their chemistry (Goldstein et al., 1981). BSEM in conjunction with EDXA is a powerful tool, providing a combination of high-resolution textural information with semi-quantitative assessment of the mineral chemistry. In this way, the majority of phases - even trace amounts of very fine-grained or cryptocrystalline minerals - can be identified. The morphology of the uranium particles was subsequently examined in SEI mode. SEI images are obtained by imaging of low-energy secondary electrons produced in, or returned from, material surfaces as a result of interaction of the high-energy electron beam with the valence electrons of atoms in the specimen surface (Goldstein et al., 1981). Image brightness and shadowing effects in SE images are dependent on the orientation of the sample surface with respect to both the incident electron beam and the electron detector. Consequently, SE images provide information on the morphology of the material being examined in the electron microscope.

3 Results

3.1 DERA SAMPLE 61/001, MORTAR AND ROOF TILE MATERIAL

3.1.1 Cement mortar debris

BSEM observations revealed fine grained particulate material rich in uranium on the surfaces of fragments of cement mortar. These were easily distinguished as bright grains against the dull, low electron backscattering coefficient calcium-silicate and aluminosilicate groundmass of the cement matrix (Figure 1). The abundance of uranium-rich particles present, estimated by counting particles from representative SEM fields of view, is of the order of 5-15 particles/mm^2. Other fine dense particulate material included mainly iron oxide and iron-calcium rich material (possibly a calcium ferrite-like phase such as brownmillerite, which may have been present in the original cement matrix.

The uraniferous particles appear to be bimodal in size distribution, and fall mainly into two ‘class sizes’ (Figure 2). The coarser material has a particle size generally of the order of 10 μm, although it varies from about 5 to 15 μm. The finer particles are typically less than 1 μm and often much less than 0.5 μm diameter. The coarser particles have a complex and variable morphology (Figures 3 and 4). These consist of composite aggregate grains, typically comprising fine flakes, plates or small blocky sub-grains of uranium-rich material and amorphous or platy sub-grains of calcium silicate or aluminosilicate material (presumably derived from the matrix of the cement). The particles are only loosely attached to the surface of the mortar particles – as is evident from the fact that they often move during observation due to charging of the sample surface under the electron beam. The finer particles appear to be simple discrete flakes or irregular grains, similar in composition to the individual component particles making up the larger composite grains.

Most particles are irregular in shape (e.g. Figure 3), although a small proportion of the composite uraniferous particles occur as more rounded aggregates (Figures 6 and 7). However, there is no evidence of uraniferous particles with the spheroidal morphology, produced by aerosolisation of DU that are reported from hard impact test firing of DU penetrators (Patrick & Cornette, 1978).

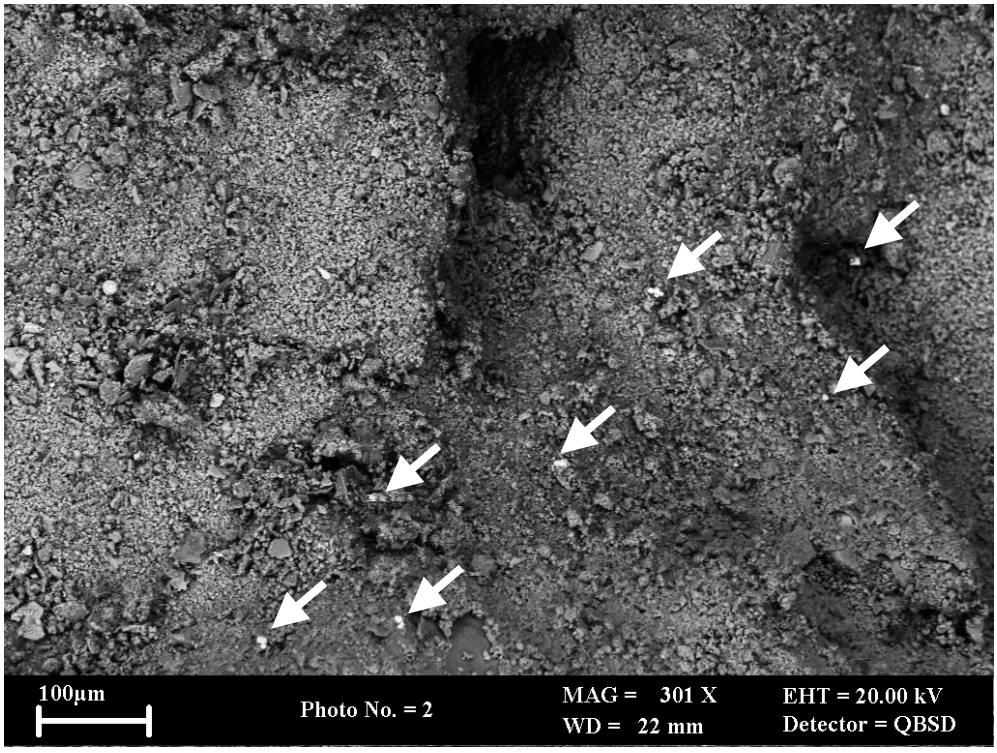

FIGURE 1. BSEM image of the surface of a fragment of cement mortar revealing the abundance of tiny particles of uranium-rich material (arrowed) resting on the cement surface, and which appear bright under BSEM imaging. DERA Sample 61/001, cement mortar.

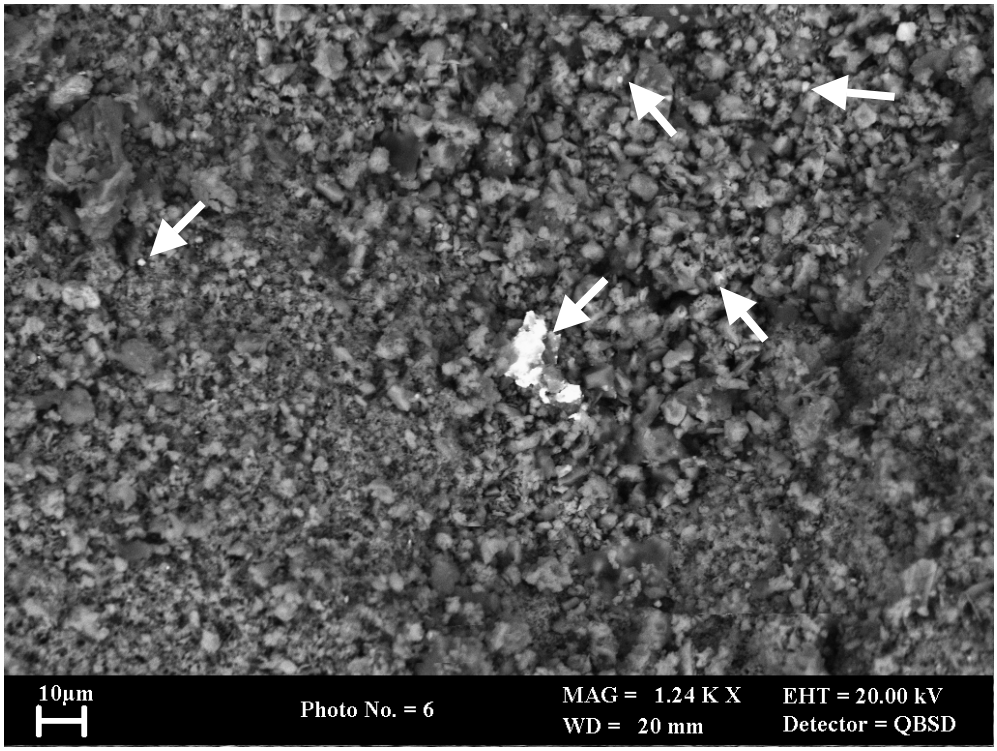

FIGURE 2. BSEM image showing bimodal variation in grain size of bright uraniferous particles (arrowed), with coarser particles on the order of 10-15 µm and finer particles <1 µm. DERA Sample 61/001, cement mortar.

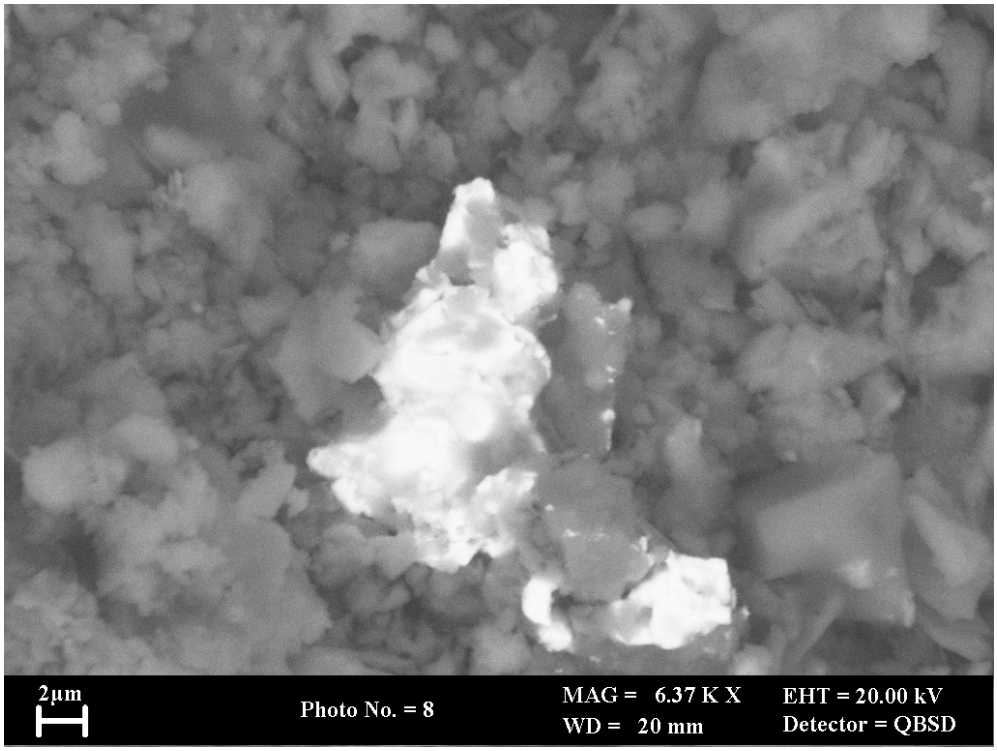

FIGURE 3. BSEM image showing complex aggregate particle comprising sub-grains of flaky uraniferous material (bright) and platy calcium silicate or aluminosilicate sub-grains. DERA Sample 61/001, cement mortar.

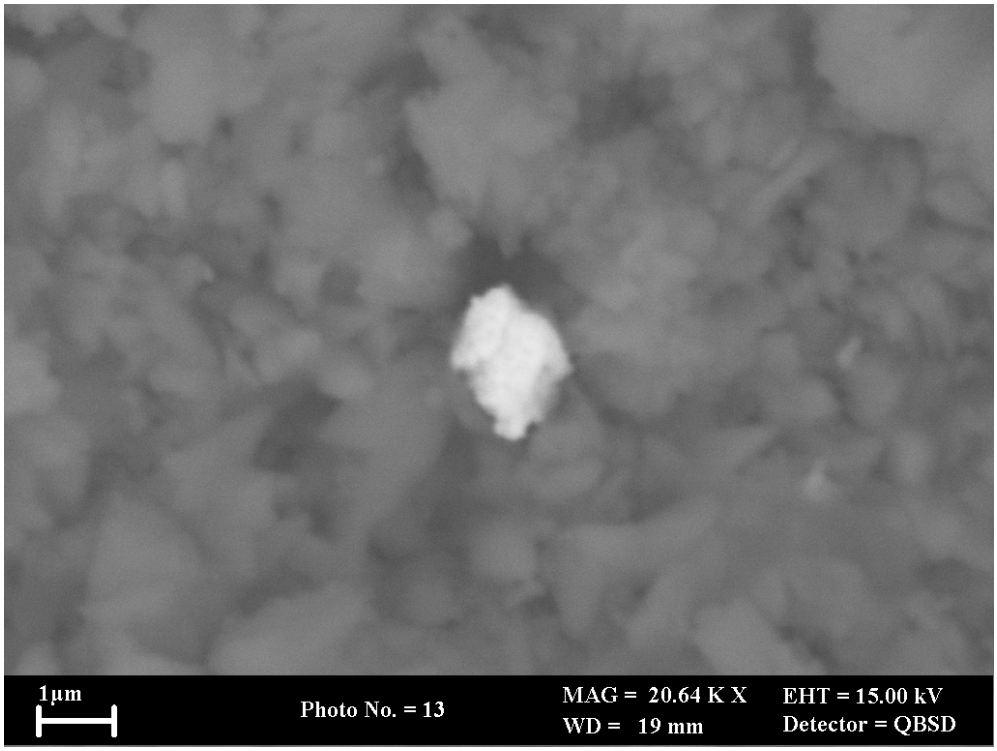

FIGURE 4. BSEM image showing fine grained and simple uraniferous particle (bright) comprising an 'aggregate' of two or three flakes of metallic uranium or uranium metal partially oxidised to uranium oxide. The morphology of this particle is shown more clearly in FIGURE 5. DERA Sample 61/001, cement mortar.

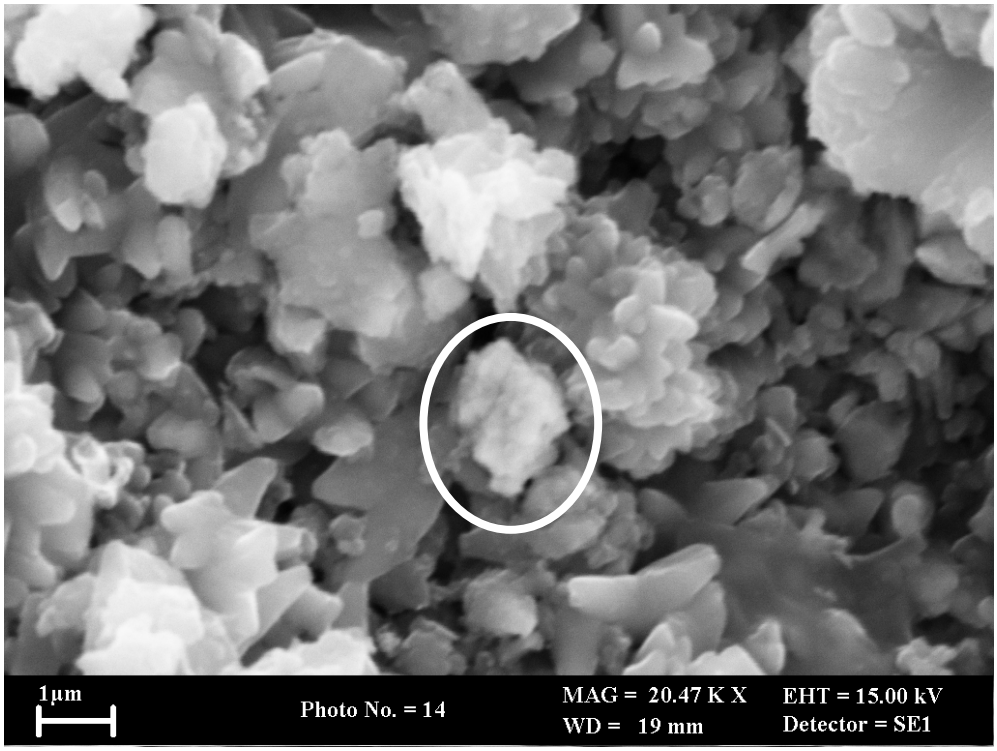

FIGURE 5. SEM image of the same field of view as seen in the BSEM image in FIGURE 4. It reveals the blocky to flaky morphology of the uraniferous particle (circled) resting on the background calcium silicate hydrate matrix of the cement mortar. DERA Sample 61/001, cement mortar.

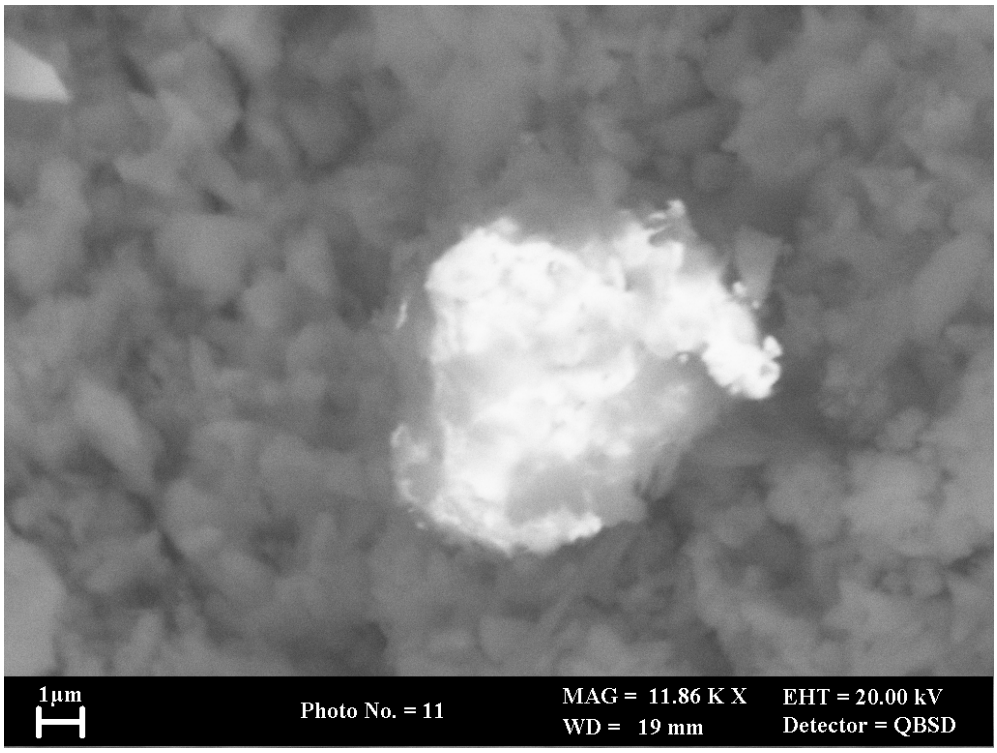

FIGURE 6. BSEM image showing bright uraniferous particle consisting of a composite grain of fine flaky uranium-rich sub-grains. DERA Sample 61/001, cement mortar.

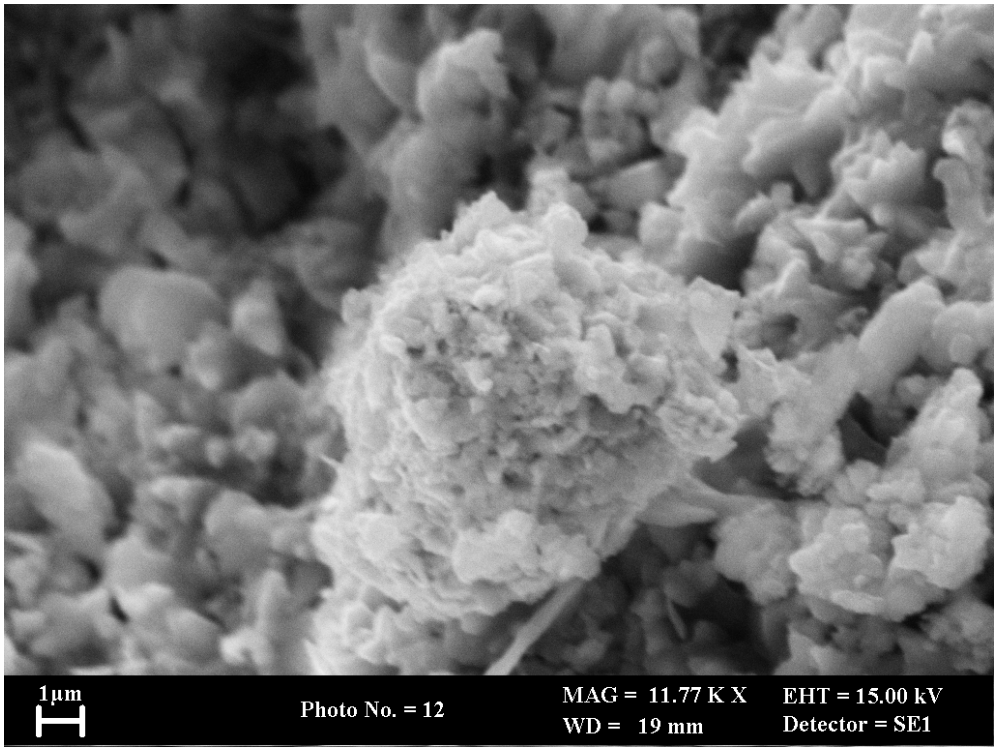

FIGURE 7. SEM image of the same field of view as seen in FIGURE 6. It shows that the aggregate uraniferous particle has a roughly sub-spherical shape. DERA Sample 61/001, cement mortar.

The composition of the uraniferous particles is variable, even within a single particle. Some particles appear to be composed mainly of uranium, calcium, silicon, aluminium and oxygen, with minor amounts of iron and titanium (Figure 8). Many of these particles are 10-15 µm in size and thus the X-ray emission detected by EDXA would be expected to be derived almost entirely from within the particles, with little contribution from the cement substrate. Often, the particles are composite particles of calcium uranium silicate or aluminosilicate sub-grains, aggregated and intimately intergrown with silica, calcium silicate and calcium aluminosilicate sub-grains. In many cases (as shown in Figure 3), BSEM reveals that bright fine-grained uranium-rich material forms fringes that 'decorate' the edges of flakes of silicate or aluminosilicate material. Earlier SEM studies of DU particles recovered from soils following test firing (Patrick & Cornette, 1978), also revealed that the DU particulate often contained iron, aluminium, silicon, calcium magnesium, potassium and titanium (elements that would almost certainly be present in soil). These authors also found that the morphology of the DU particulate in the soil was completely different to globular or spherical particles produced by aerosolisation. However, Patrick & Cornette (op cit.) attributed the chemistry of these particles to contamination effects. Whilst it is likely that the cement substrate contributes to the EDXA signal in the case of the fines particles observed in the Kosovo sample (as discussed below), the similarity in the composition obtained from different areas of larger grains suggests that calcium, silica and aluminium detected by EDXA are an intrinsic part of the larger uraniferous particles. Studies of natural combustion-metamorphism of uraniferous rocks (Alexander, 1991) have shown that uranium-rich material can react with calcium and silicate minerals at high temperatures to form complex phases, including calcium-uranium oxides. The compositions and morphology of these particles observed in DERA Sample 61/001 strongly suggests that DU material has reacted (at least partly) with the calcium aluminosilicate cement mortar matrix. The decoration of the edges of calcium aluminosilicate flakes by very fine uranium-rich material (e.g. Figure 3) possibly suggests that the uranium-rich particles formed by a process of 'condensation' of vaporised DU with nucleation on silicate particles.

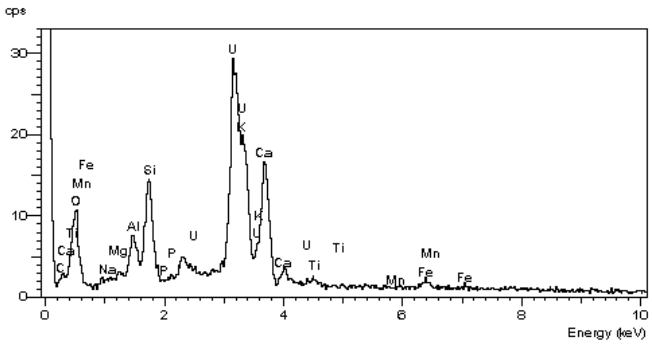

FIGURE 8. EDXA spectrum of DU particulate material from the surface of a fragment of cement mortar. The particle is composed of uranium, calcium aluminosilicate.. DERA Sample 61/001, cement mortar.

Other particles, are much more uranium-rich, containing either uranium only (with small amount of titanium), or uranium and oxygen, with a small and usually very variable concentration of silica, aluminium and calcium (Figure 9). Such particles are typically less than 5 μm in size. They possibly represent small particles of partially-oxidised uranium metal and uranium metal, with a contribution of calcium, silicon and aluminium, from the cement matrix in the EDXA analysis (due to excitation of X-rays from a volume larger than the particle of interest, cf Goldstein, 1981).

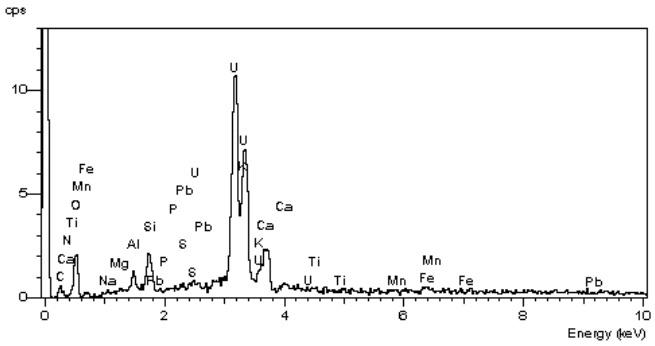

FIGURE 9. EDXA spectrum of DU particulate material from the surface of a fragment of cement mortar. The particle is composed mainly of uranium with minor contamination of the EDXA signal due to X-ray emission from the calcium aluminosilicate in the cement substrate. DERA Sample 61/001, cement mortar.

3.1.2 Fired-clay roof tile debris

BSEM observations revealed that the surfaces of fired-clay tile fragments were also covered with fine uranium-rich dust (Figure 10). The DU particles are presumably derived from the settling of DU dust derived from DU rounds since uranium-rich material is not present on freshly-fractured tile surfaces. Some areas of the tile surface examined contained up to 10 particles/mm^2.

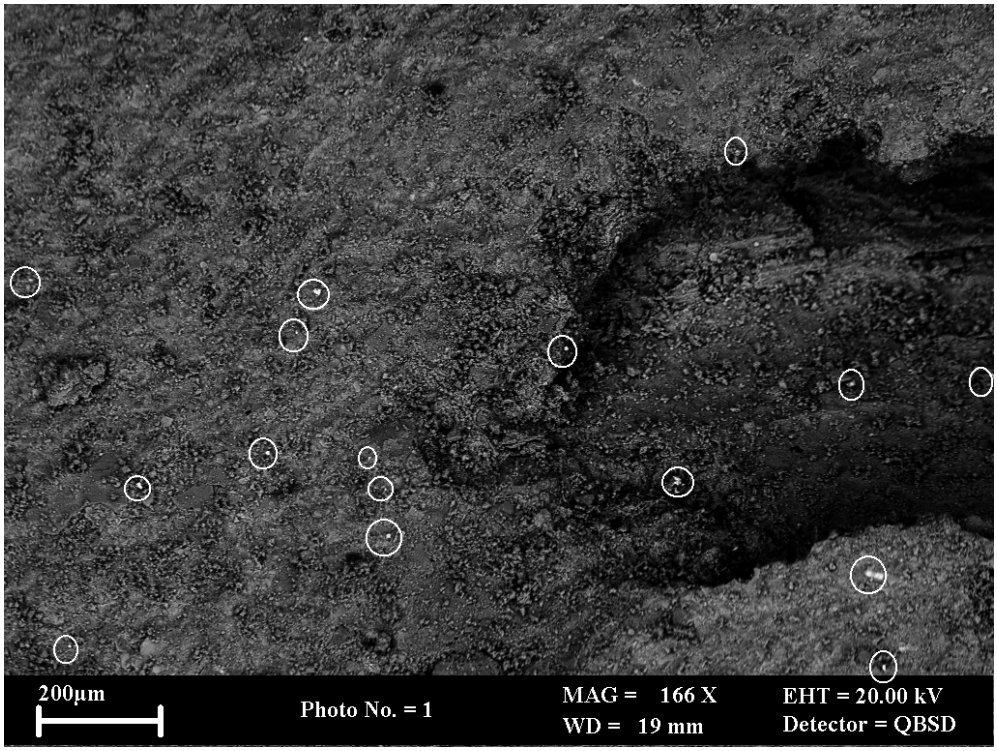

FIGURE 10. BSEM image with contrast set to show the distribution of fine bright uraniferous particles (circled) on the 'natural' surface of a fired clay tile fragment. DERA Sample 61/001, fired-clay tile fragment.

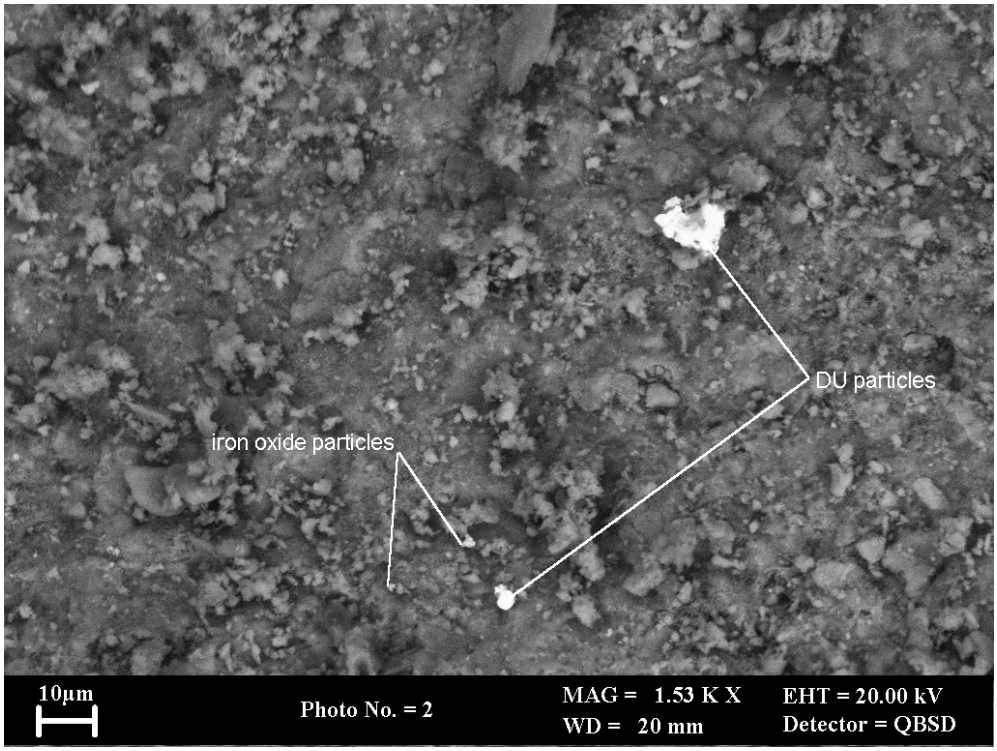

FIGURE 11. BSEM image illustrating the two size categories of uraniferous particulate (bright) found on the 'natural' surface of a fired clay tile fragment. DERA Sample 61/001, fired-clay tile fragment.

As with the cement mortar fragments, the uraniferous particles fall into two size categories: particles varying between 5 to 15 μm diameter; and particles <2 μm (and generally <1 μm) diameter (Figure 11). The uranium-rich particles are generally flaky in morphology. Again, this is different to the spheroidal morphology reported for DU aerosols produced in test firing (Patrick & Cornette, 1978). Some uraniferous particles appear to be spongy and 'turned' flakes that may represent oxidised or partly-oxidised fine uranium metal 'swarf' or 'shards'. EDXA supports this, with X-ray spectra indicating that the particles contain mainly uranium or (uranium and oxygen), with small amounts of silicon, aluminium and iron in similar peak ratios to that of (and therefore probably contributed by) the background fired-clay (Figure 12). Other particles of similar composition appear to be relatively coarse (up 15 μm) aggregates of fine (<1 μm) uranium-rich sub-grains (Figures 13 and 14).

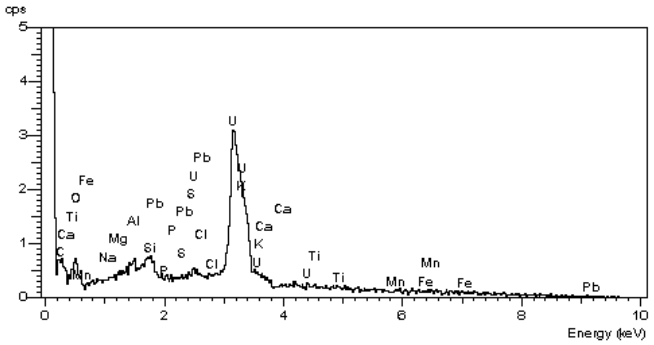

FIGURE 12. EDXA spectrum of uraniferous particulate from surface of a fired-clay tile fragment. It appears to be composed mainly of uranium, with oxygen, aluminium and silica probably representing background X-ray emission from the tile substrate. DERA Sample 61/001, fired-clay tile fragment.

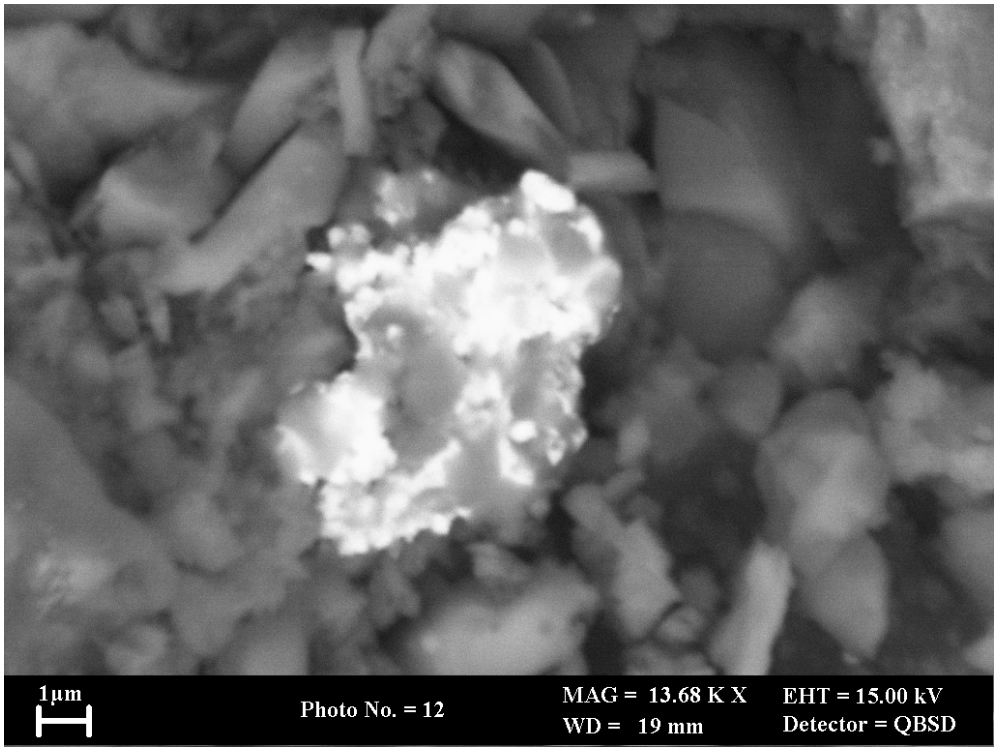

FIGURE 13. BSEM image uraniferous particulate (bright) comprising an aggregate of fine sub-grains. DERA Sample 61/001, fired-clay tile fragment.

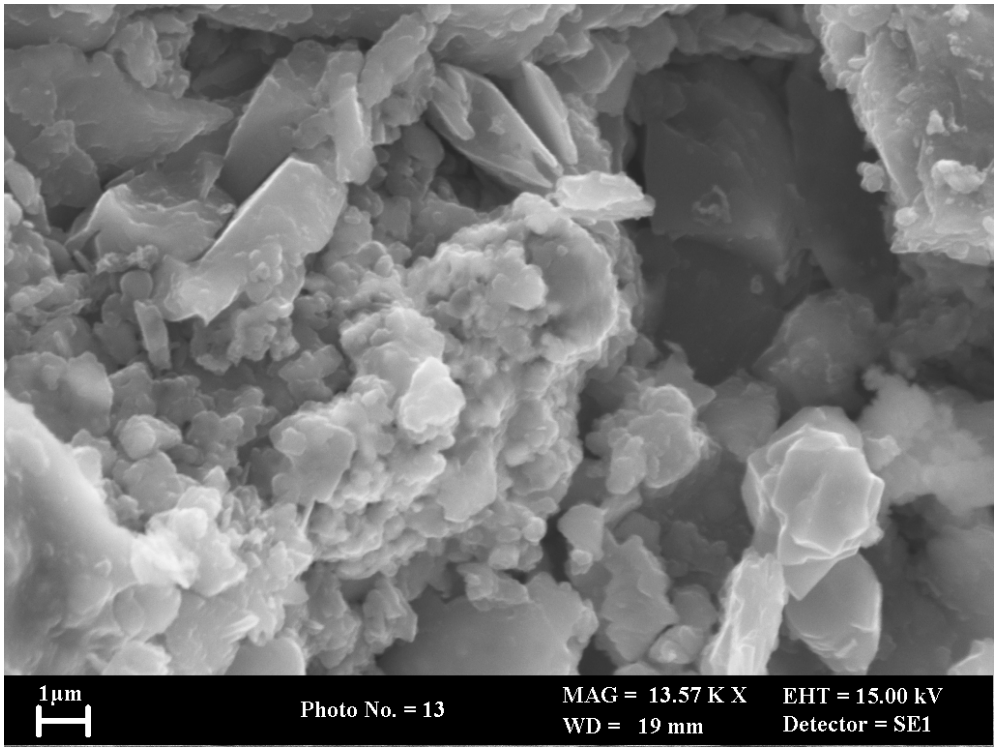

FIGURE 14. SEM image of the same area as FIGURE 13, illustrating the granular sub-grain aggregate morphology of the coarser uraniferous particulate found on the 'natural' surface of a fired clay tile fragment. DERA Sample 61/001, fired-clay tile fragment.

High carbon contents were found in some of these uraniferous particles. This suggests that they may have reacted to form uranium carbonate phases.

3.2 DERA SAMPLE 61/003, CONCRETE FINES MATERIAL

Uraniferous particulate is abundant on the surfaces of cement fragments in this sample. In some cases, over 100 particles/mm^2 were found on some cement grains (Figure 15). This is consistent with the higher surface radioactivity measured from DERA Sample 61/003 (Section 2.1).

The uraniferous particles in this sample have a similar size range to those observed in DERA Sample 61/001. Smaller particles (up to 2 µm) tend to occur as flakes. However, coarser particles are composite, and comprise aggregates of fine uraniferous material, often intergrown or admixed with calcium silicate or aluminosilicate phases (cf. Section 3.1.1). Detailed BSEM observations reveal that many of the particles have formed by nucleation of very fine uranium-rich material around 'cores' of flaky calcium aluminosilicate or calcium silicate (Figure 16). The uraniferous material is composed of uranium and oxygen (probably uranium oxide or hydroxide) or a more complex calcium-uranium-silicon oxide phase. In some particles the uranium-rich rim may even replace the silicate 'core'. This implies that the DU has reacted to some extent with the calcium silicate cement matrix of the concrete, and is similar to the observations on DU particulate in the cement mortar in DERA Sample 61/001 (as described previously in Section 3.1.).

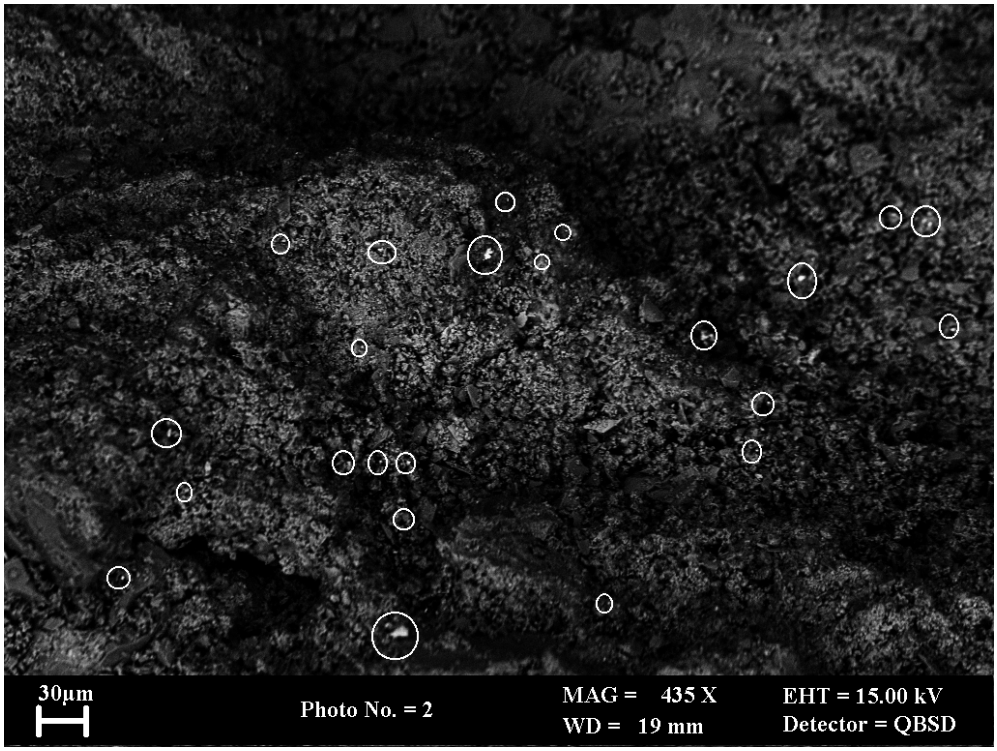

FIGURE 15. BSEM image the abundance of uraniferous particulate (bright, circled) found on the surface of cement fragment. DERA Sample 61/003, Concrete debris.

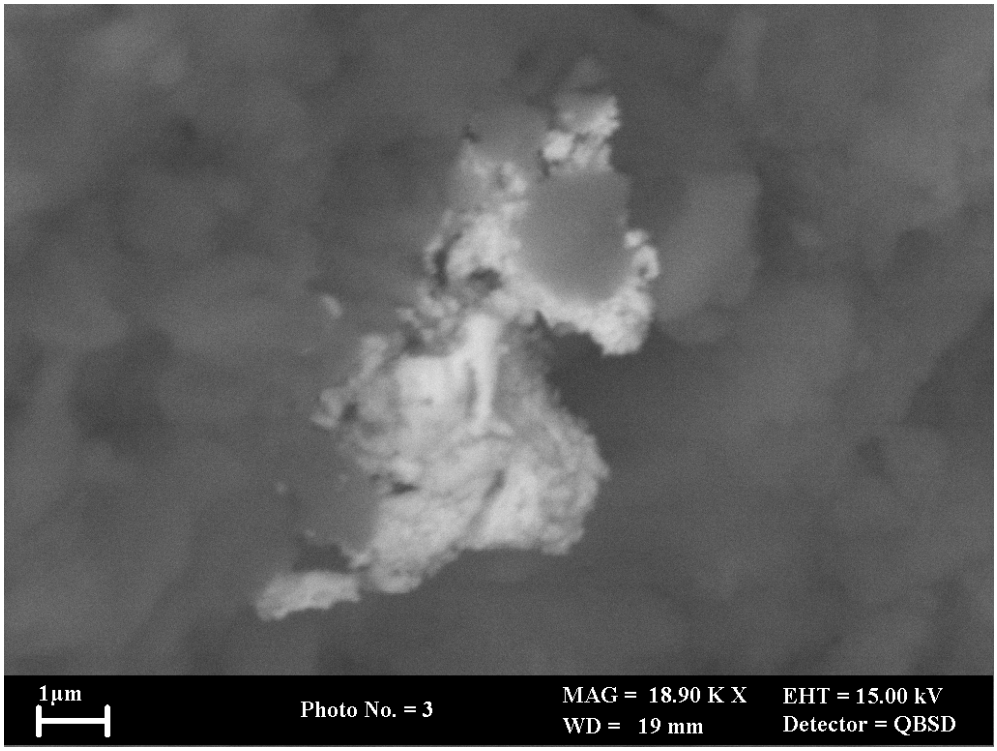

FIGURE 16. BSEM image illustrating the morphology of a large uraniferous particle resting on the surface of a cement fragment. The bright uraniferous material appears to be an admixture of uranium oxide and a calcium-uranium-silicon-oxide phase, and has a microgranular texture. This uraniferous material appears to have nucleated around flakes of calcium silicate (dull grey) DERA Sample 61/003, concrete debris.

4 Summary

Two samples of building debris from the VJ Barracks in Kosovo (DERA Samples 61/001 and 61/003) were examined by SEM techniques to try to identify the nature of potential contamination by DU derived from armour-piercing DU rounds used by NATO aircraft against the building. The samples were collected 'as grab samples' by MoD/DERA representatives and examined by BGS. The main observations and conclusions are summarised below

1. BSEM imaging proved very successful in locating dense uranium particulate on the surfaces of masonry fragments (cement mortar, concrete dust and fired-clay roof tile) from both samples. The uranium-rich particles are concluded to have originated as dust from the DU munitions impacts because no similar uraniferous material was found within freshly broken surfaces of the masonry material.

2. The uraniferous particulate material falls into two size classes:

   (i) very fine material with a particle size less than 2 μm and generally less than 0.5 μm;

   (ii) coarser particles, typically 10-15 μm in size, which comprise aggregates of finer uraniferous material with calcium silicate or aluminosilicate material.

3. None of the uraniferous particles observed illustrated the typical spherical morphology of DU particulate reported previously from test firing of DU munitions against hard targets (cf. Patrick & Cornette, 1978). Such particles might be expected to have formed by rapid cooling of aerosolised molten metal. Instead, the uranium particles identified in building debris from material adjacent to impact sites at the VJ Barracks, are largely flaky in morphology (single particles) or aggregates of flaky or diffuse material.

4. The uraniferous particulate displays a variety of compositions. Some material appears to be oxidised or partly oxidised microscopic uranium metal shards. This is particularly the case for particles found on the tile fragments. However, particles found on cementitious debris (either cement mortar or concrete dust) are more complex. These particles vary from possible uranium oxide to calcium-uranium silicate or calcium-uranium aluminosilicate phases. Although it is possible that the EDXA analyses obtained from very fine uraniferous particulate were influenced by background X-ray emission from the cement substrate, observation of coarser particles (>10 μm) suggest that the calcium, silicon (and aluminium) are intrinsic to the uraniferous particle itself. This implies that there has been reaction between the uranium and calcium silicate or calcium aluminosilicate matrix of the cement. There is also evidence that some of the uraniferous particulate has altered to a uranium carbonate phase. This was observed in particles found on tile fragments, and which contained high carbon contents indicated by EDXA. Since these observations were made on SEM mounts which were not carbon-coated, it must be concluded that the carbon is intrinsic to the uraniferous particles.

References

Most of the references listed below are held in the Library of the British Geological Survey at Keyworth, Nottingham. Copies of the references may be purchased from the Library subject to the current copyright legislation.

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