



# Formation of Metal Nanoparticles Directly from Bulk Sources Using Ultrasound and Application to E-Waste Upcycling.

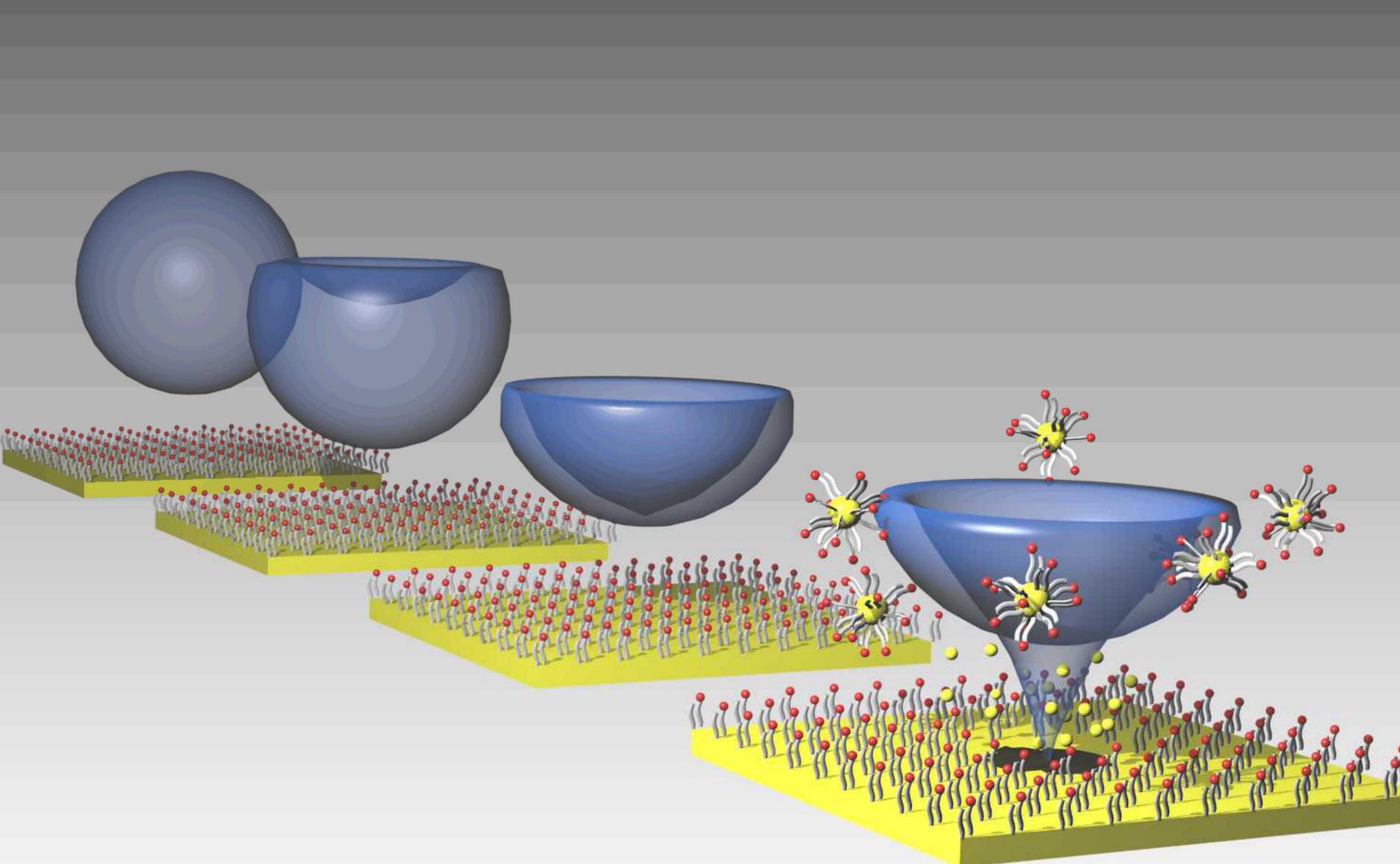
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## Introduction

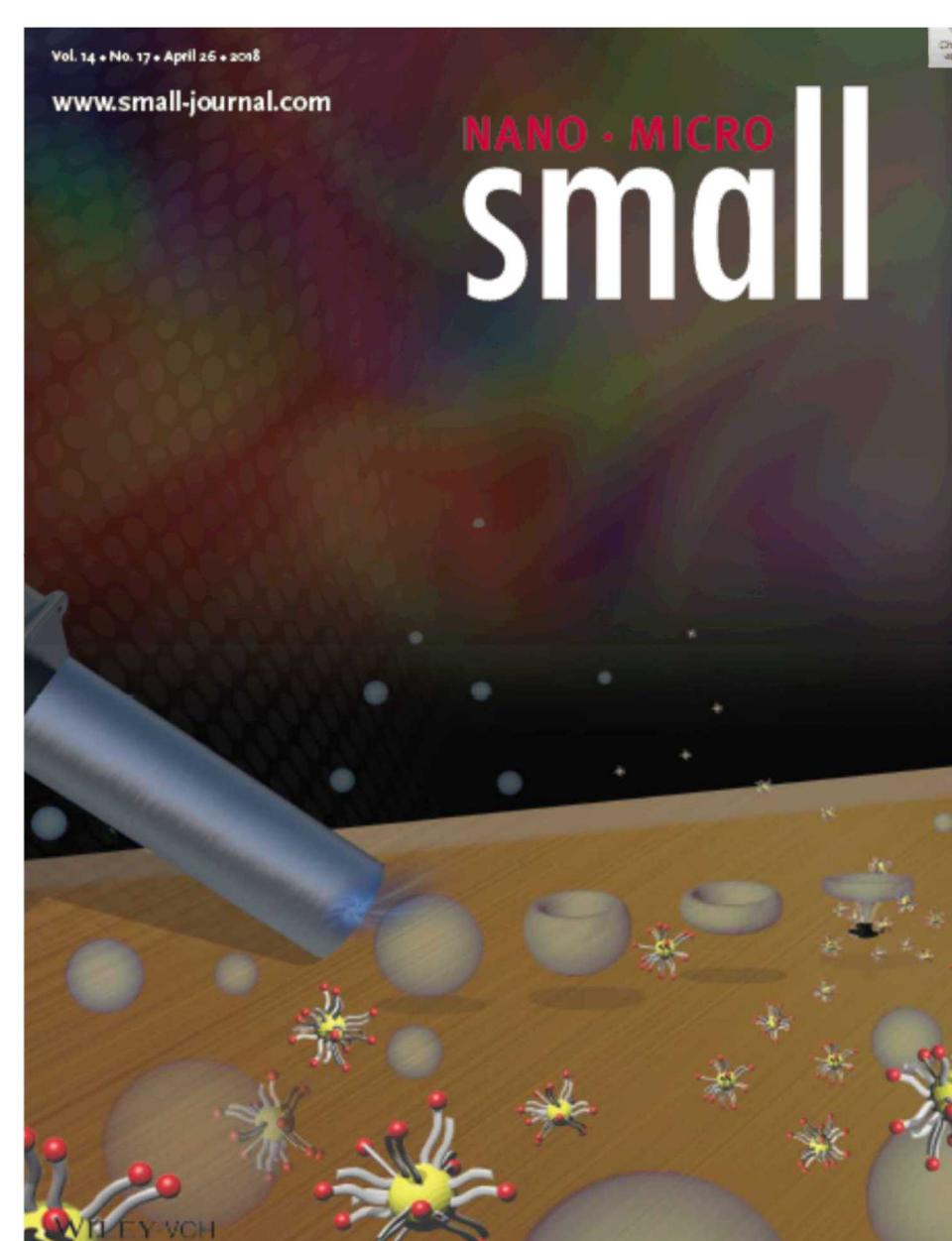
A method for creating nanoparticles directly from different types of bulk metal was developed. The process occurs in water and was shown to form nanoparticles of gold, silver, nickel and their alloys. These high value materials can be expensive to produce by traditional methods and often require potentially toxic chemical intermediates.

Ultrasonication forms cavitation bubbles which collapse on the metal surface impacting violently and ejecting material, the same effect that degrades marine propellers. The ejected material is captured by an organic bilayer, which stabilize it in the form of nanoparticles. Nanoparticles could be formed from bulk metal regardless of initial form factor. We then form nanoparticles from the gold contact of cellular SIM cards, meaning the method is useful for the recovery of gold from E-Waste, a potential \$22.2 billion source.

## Mechanism



Schematic representation of cavitation bubble collapse leading to pit formation and material ejection. When bulk gold is subjected to ultrasonication in the presence of DDAB in water an organic bilayer forms on the surface. This allows material that is ejected due to cavitation erosion to be stabilized as nanoparticles in solution.

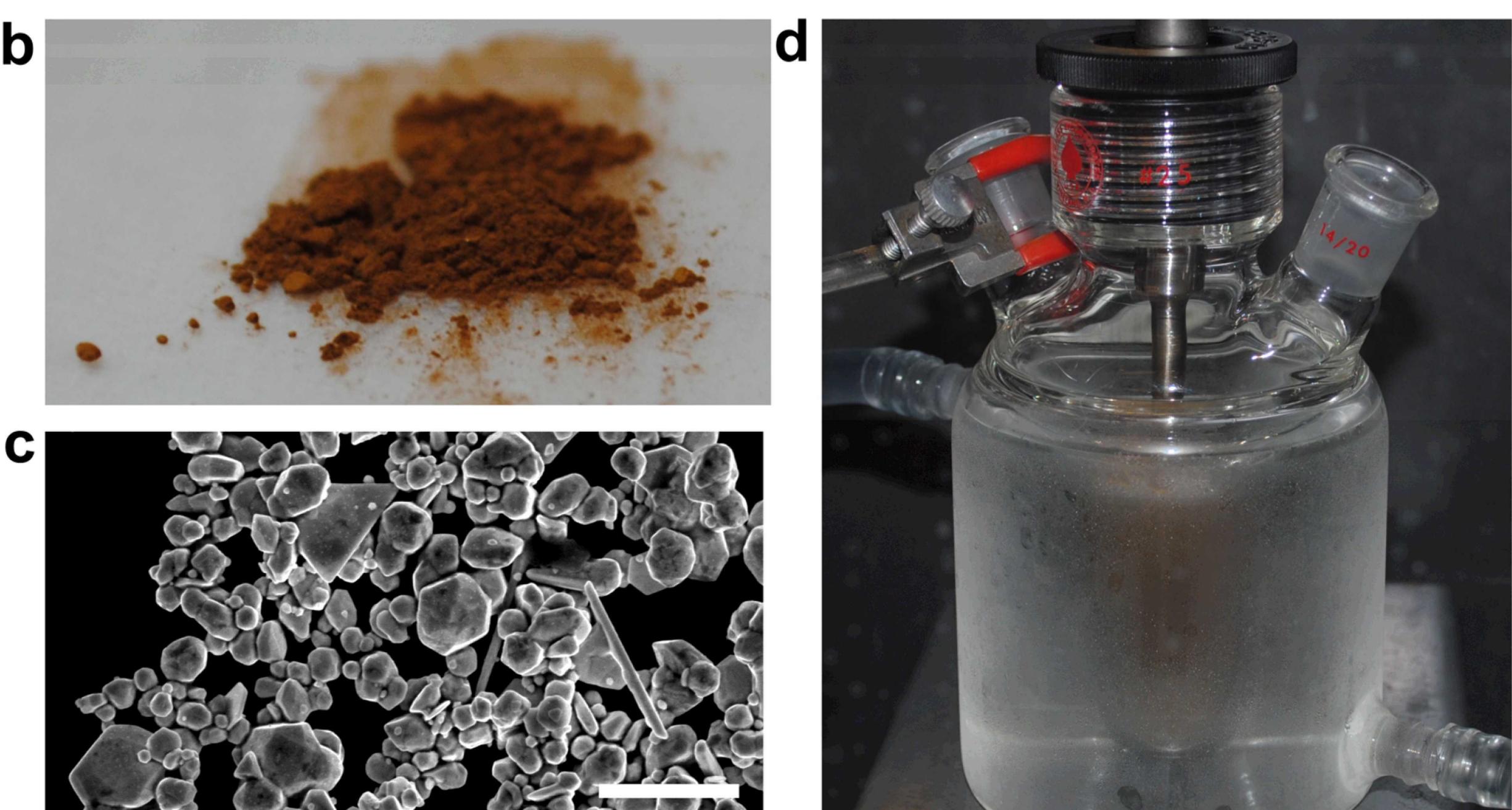


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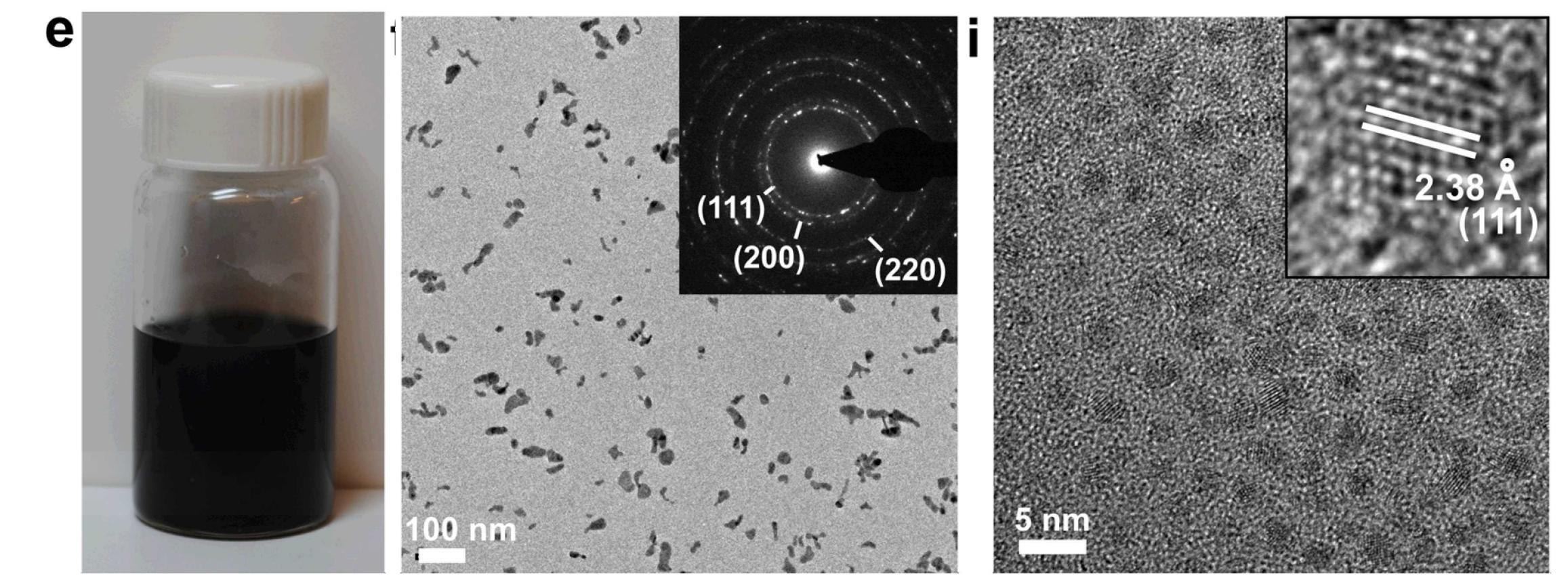
This work was performed, in part, at the Center for Integrated Nanotechnologies, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science. Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA-0003525. This paper describes objective technical results and analysis. Any subjective views or opinions that might be expressed in the paper do not necessarily represent the views of the U.S. Department of Energy or the United States Government.

## Gold Nanoparticle Formation

Bulk gold in the form of a powder, which SEM analysis revealed to consist of hexagonal and plate like particles is placed in an ultrasonication reaction setup consisting of a glass-cooling jacket and conical shaped reaction vessel. Ultrasonic treatment is carried out by a 6.4 mm diameter Ti sonication tip operating at 18 W and 20 kHz for up to 6 h at 0 °C.



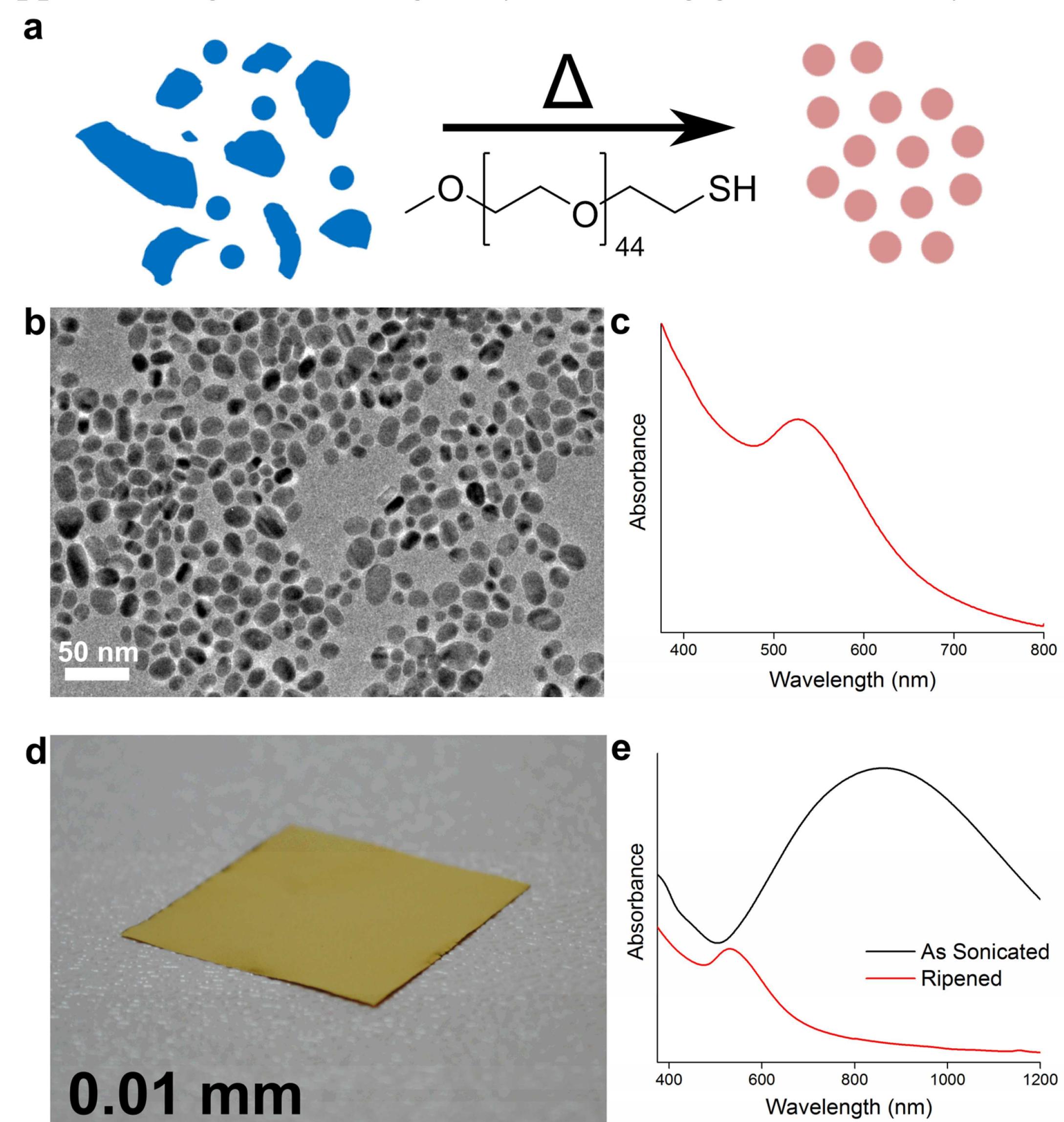
A blue solution formed which is characteristic of finely divided gold. TEM revealed nanoparticles *fcc* Au nanoparticles.



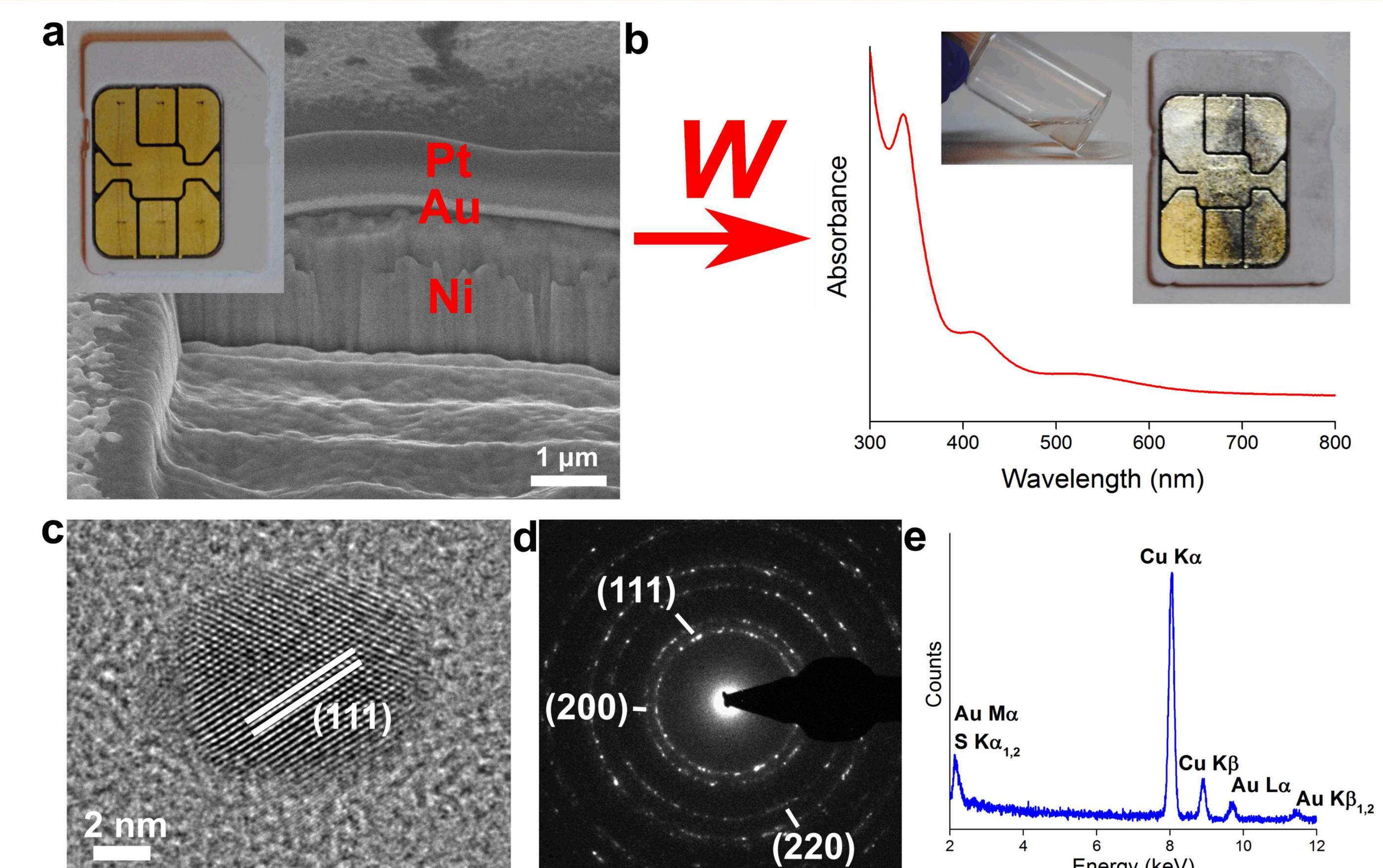
## Digestive Ripening

For effective application of gold nanoparticles a well-defined surface plasmon resonance is typically required. Therefore, we employed a digestive ripening step using a recyclable thiolated polymer (PEG-2000-SH). This was performed in water and resulted in a plasmon resonance at  $\lambda_{\max} = 530$  nm.

We showed that nanoparticles could be formed regardless of the form factor of bulk gold e.g., gold foil. The method could also be applied to Ag, and Au-Ag alloy indicating good versatility.



## E-Waste Upcycling



The method was shown to be capable of stripping the vast majority of gold from electronic waste, evidenced by the disappearance of the gold color on a cellular SIM card, and directly forming nanoparticles. FIB milling experiments revealed that the surface of the SIM card consists of a thin Au layer supported on a Ni substrate. A UV-vis spectrum of the resulting solution after ultrasonication of a SIM card in a mixture of water, DDAB, and dodecanethiol shows a lightly colored pink color, characteristic of gold nanoparticles.

TEM and selected area diffraction (SAD) revealed the formation of gold nanoparticles with the *fcc* crystal structure. Energy dispersive X-ray spectroscopy (EDS) and showed that Au was the only metal present in solution (Cu signal is due to the TEM grid). Ni nanoparticles were also formed confirming the versatility of the method, however these were removed from solution using a magnetic field.