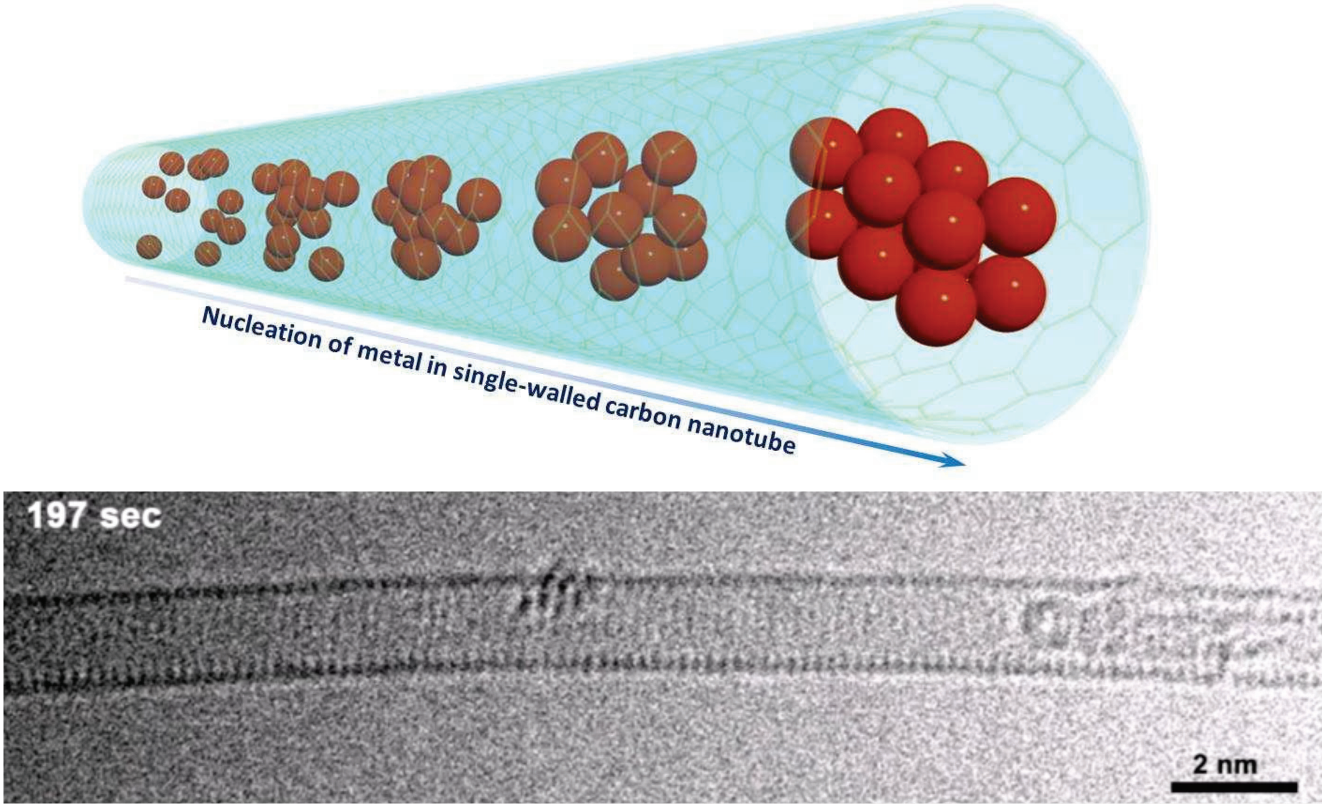

Figure 1: The schematic drawing shows how iron atoms assemble into an ordered crystal lattice structure. (top). TEM image of a film showing the crystallization of individual atoms in a carbon nanotube in detail (bottom).

Crystallization: Atomic Lego or Particle Chaos?

Crystallization of metals filmed

September 1st, 2020 - If several iron atoms come together, they form an ordered crystal structure. How this works in detail has now been clarified by an international team of researchers with the help of live recordings of the crystallization. This solved the discussion about two contradicting theories about the nucleation of crystals.

Crystals are known from the kitchen in the form of sugar and table salt, or from the winter front garden, which is covered by ice crystals. And in metals, too, the tiny atoms are regularly arranged in what is known as a crystal lattice. Metals and their behavior under changing influences have been studied quite well, but so far it has not been proven how the nucleation of crystals takes place: how individual atoms begin to form a three-dimensional lattice structure.

Researchers from the materials science electron microscopy group at Ulm University, in cooperation with colleagues from England and Japan, have now succeeded in observing precisely this “hour of birth” of a crystal. In doing so, they were breaking new ground. “In the standard literature so far there have been two models of how a crystal could form. One assumed that atoms, like Lego bricks, put together one after the other and thus build the crystal lattice. The second model assumed that there could be a disordered intermediate phase from which the crystal is formed, ”explains Professor Ute Kaiser, head of materials science electron microscopy at Ulm University. Kaiser’s team has now proven which crystal formation model applies.

Crystallization in carbon nanotubes

It was initially a coincidence that the microscopy specialists were able to watch a crystallization live. For the originally planned investigation, the colleagues from Nottingham had introduced iron atoms into so-called carbon nanotubes. These are microscopic tubes, the walls of which are exactly one carbon atom thick, and which serve as nano test tubes, as the researchers explain. When looking through the “Titan” electron microscope, which has been corrected for defects, the Ulm scientists witnessed how the individual iron atoms clustered together - with a resolution of one image per second, i.e. practically in real time.

How many atoms does it take to nucleate?

The nucleation was triggered by the energy transfer of the electron beam from the microscope to the iron atoms. And it was revealed that initially a few iron atoms formed an amorphous phase, i.e. a liquid-like accumulation of atoms without an internal structure (Figure 1).

“We found out that the atoms only begin to arrange themselves into a regular lattice structure above a critical number between 10 and 20. We were thus able to provide proof that the nucleation of crystals is based on a two-stage nucleation mechanism (Figure 2). ”describes Dr. Kecheng Cao the discovery of a transition phase in the formation of metallic crystal structures. The first author of the current study is a postdoc in the ma-

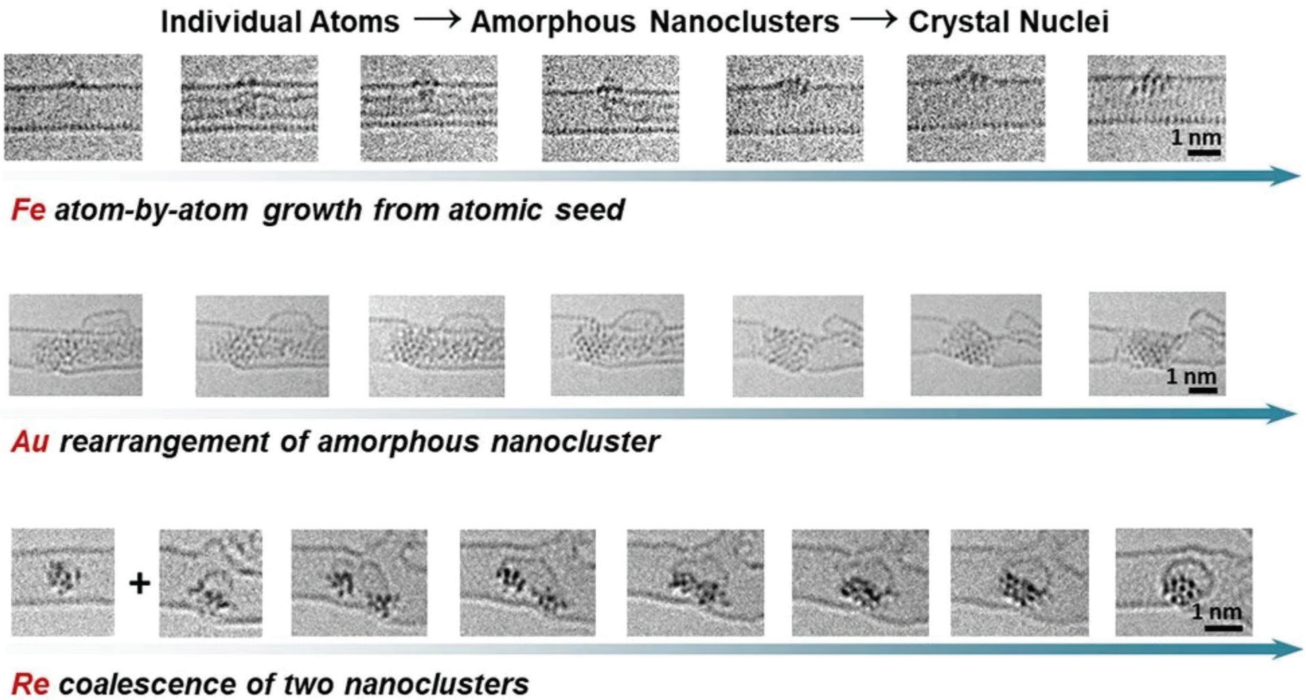

Figure 2: The compilation of TEM images shows the nucleation of iron, gold and rhenium atoms: from the individual atom through the phase of amorphous nanoclusters to the ordered crystallization nucleus.

terials science electron microscopy department at Ulm University.

Alloys are the next step

The scientists also observed the crystallization process in tests with gold and rhenium atoms as well as further tests with iron and always saw similar behavior. And what's next? "However, this groundbreaking observation does not mean the end of our research. Because with other materials there could be different processes. We therefore also want to examine more complex materials such as metal alloys for their crystallization behavior, " says Prof. Kaiser.

The new "SALVE" microscope will be used here. The low-voltage transmission electron microscope developed at the University of Ulm, with two image aberration corrected, is one of the most powerful devices of its kind worldwide. It has a resolution three times higher than current single-corrected TEM and allows much deeper insights into the world of atoms .

Original publication

Kecheng Cao, Johannes Biskupek, Craig T. Stoppiello, Robert L. McSweeney, Thomas W. Chamberlain, Zheng Liu, Kazu Suenaga, Stephen T. Skowron, Elena Besley, Andrei N. Khlobystov & Ute Kaiser: Atomic Mechanism of Metal Crystal Nucleus Formation in a Single-Walled Carbon Nanotube, Nature Chemistry (2020), DOI: 10.1038/s41557-020-0538-9

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