

## Nanocontact and nanowire formation between macroscopic metallic contacts observed by scanning and transmission electron microscopy

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Transmission and scanning electron microscopy fitted with a chemical analysis reveal that connections between two macroscopic metallic (gold or copper) wires do not break cleanly but are stretched into many filaments of nanometer size leading to nanowire formation. [S0163-1829(97)12307-4]

Recently, it has been observed that the current flowing between two household wires vibrating in and out of contact exhibited conductance quantization (CQ) at room temperature<sup>1</sup> (with the spin degeneration quantum  $2e^2/h$ , where  $e$  and  $h$  are the electron charge and the Planck constant, respectively). The reason for this was proposed to be that when the contact between the wires breaks or forms, it is not done in a clean way, but stretches into nanofilaments.<sup>1</sup> Analogously, CQ has been observed in a STM configuration<sup>2-4</sup> and by breaking controlled junctions. In these cases, it is also assumed that having STM microcontacts pulled apart, at the end should form a nanofilament that quantizes the current.<sup>5,6</sup> Molecular-dynamics simulations<sup>7,8</sup> show that when a gold contact is stretched a nanowire is formed. However, such nanowires or nanofilaments have not been physically observed yet.

In this paper, we present experiments in which the structure of nanocontacts between two macroscopic metallic wires is observed by transmission (TEM) and scanning electron microscopy (SEM). The chemical composition of the contacts is revealed by electron dispersive x-ray spectrometry (EDX).

Imagine two household wires twisted together in a helix as depicted in the SEM micrograph [Fig. 1(a)]. As time passes, one would expect that the torque will break some of the contact areas. The structure of the slightly broken contacts can be investigated by SEM and TEM. The metallic wires diameter is 0.25 mm and 0.025 mm, respectively, for SEM and TEM experiments. In order to verify that the nanocontacts observed between the two macroscopic metallic wires are not artifacts, different metals have been used in our experiments: gold and copper. Our SEM and TEM investigations have been carried out using, respectively, a Cambridge Stereoscan 250 Mk2 fitted with an ultrathin window energy dispersive x-ray spectrometer and a 200 Kv JEOL 2000FX.

Figure 1(b) shows a magnification of a contact area with a “bridge” between the two wires clearly seen. In this image, the “bridge” is seen to be itself broken into two large threads of  $\approx 400$  nm width, each one in itself presenting a fibrous structure [the smallest feature we can resolve is  $\approx 200$  nm, see arrow 1 in Fig. 1(b)] and also small hairline fractures: the “bridge” is composed of nanocontacts. In order to determine the chemical composition of the “bridge,” we

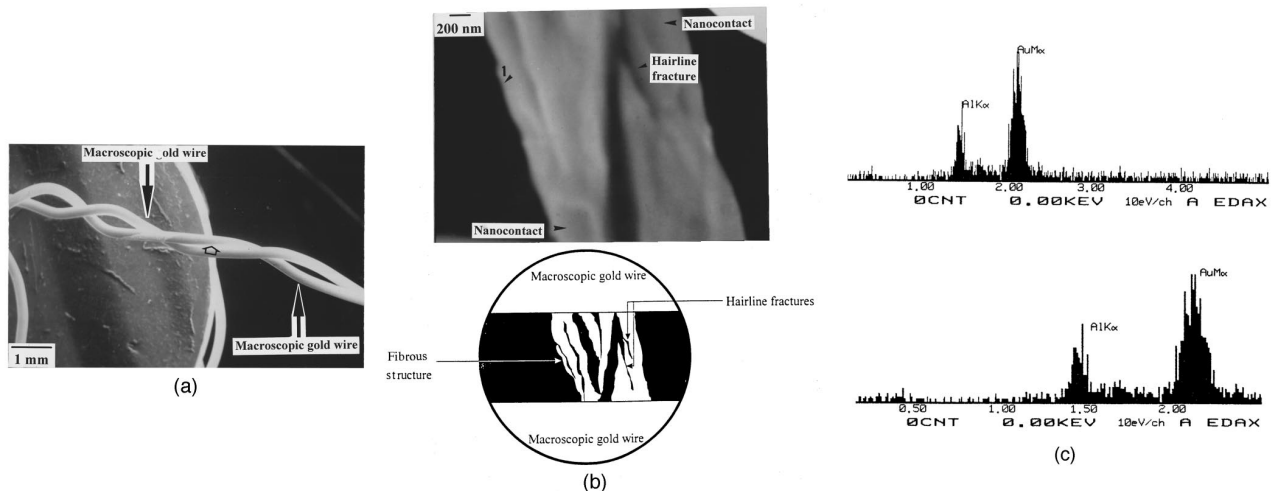


FIG. 1. (a) SEM image showing the two macroscopic gold wires twisted together in a helix. The arrow indicates the region studied. (b) SEM image showing clearly the morphology of a contact acting as a “bridge” between the macroscopic wires: this one is composed of nanocontacts. The bridge is broken into two large threads ( $\approx 400$  nm wide), each one presenting in itself a fibrous structure (the smallest feature we can resolve is  $\approx 200$  nm). Notice also the presence of small hairline fractures. (c) EDX chemical analysis performed on this bridge showing a large Au signal. The other signal (Al) is due to the sample holder.

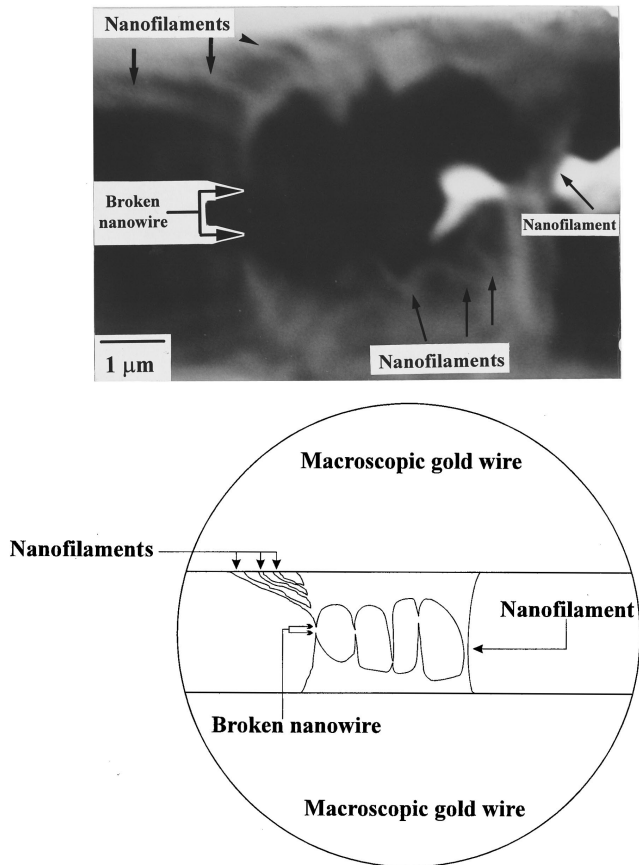


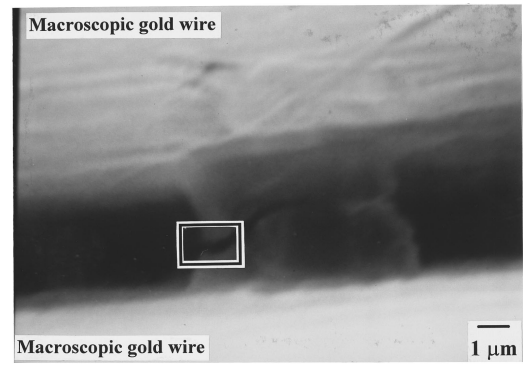
FIG. 2. SEM image showing another membrane bridge presenting nanofilaments between the two macroscopic gold wires.

have performed an analysis of this one using an ultrathin window energy dispersive x-ray spectrometer. A large Au signal has been obtained as shown in Fig. 1(c) (the aluminum signal comes from the sample holder) but not any other contaminants as oxygen, carbon, etc. The probe size used during microanalysis was  $\approx 250$  nm for gold detection.

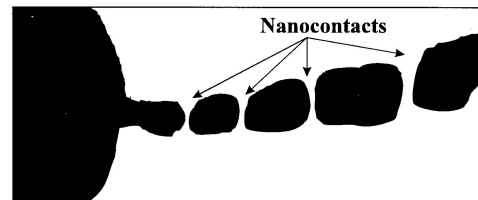
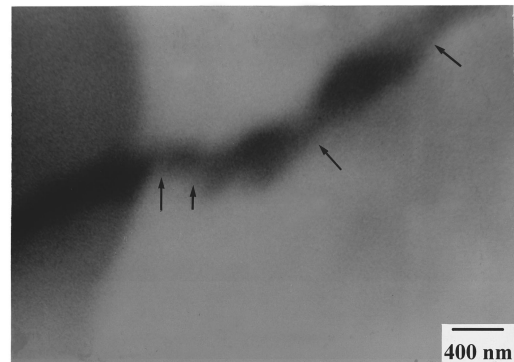
This is not the only manifestation of the narrow-filament formations; there are many other configurations that we observed by SEM leading to the stretched structure during the contact breaking. Another example is presented in Fig. 2 where now we have looked at the filamentlike structure of another broken contact. In this case, we can observe nanofilaments (see arrows in Fig. 2) and a broken nanowire between the two macroscopic gold wires.

Another example is shown in Figs. 3(a)–3(e), where a kind of membrane bridge has been formed between the wires [Fig. 3(a)]. A magnification displayed in Fig. 3(b) shows clearly four nanocontact formation in the range of 100 nm [see arrows in Fig. 3(b)]. Probably, they are smaller; however, the low contrast obtained in SEM images due to their thinness does not allow us to obtain a better resolution in this case. Finally, as time passes the nanocontacts depicted in Fig. 3(b) are broken as shown in Fig. 3(c). EDX analysis has been also performed on this other configuration revealing gold.

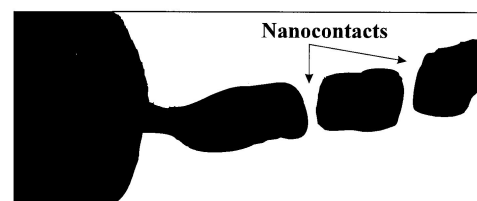
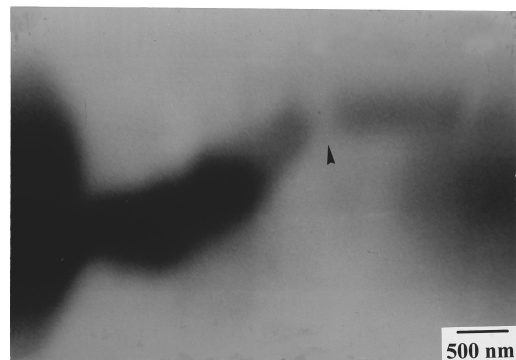
The SEM experiments have been reproduced using two macroscopic copper wires in order to verify that the gold nanocontacts observed are not artifacts. Figure 4(a) shows clearly another kind of membrane “bridge” between the



(a)

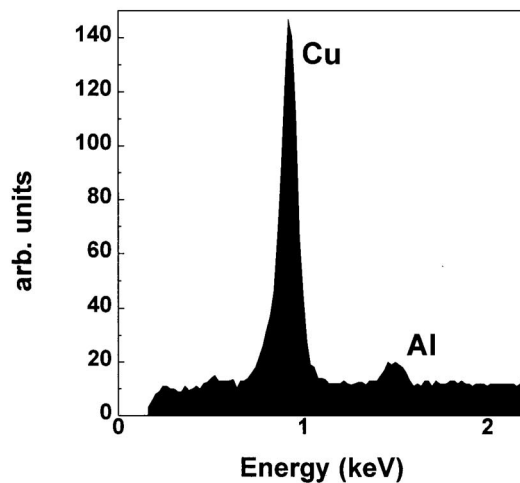
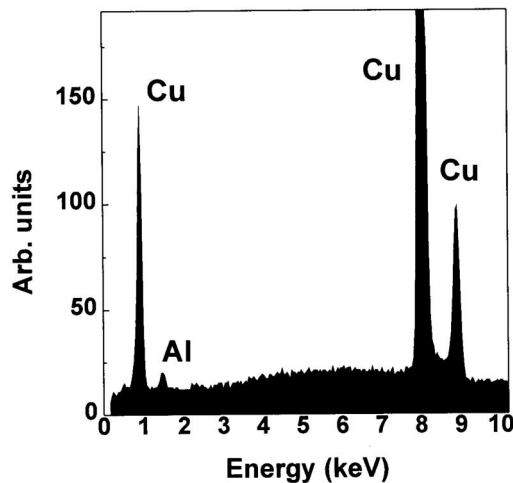
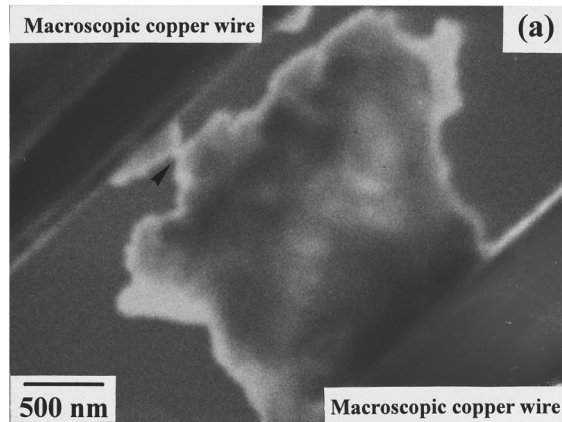


(b)



(c)

FIG. 3. (a) SEM image showing another nanocontact configuration, a kind of membrane bridge. (b) Magnification of the selected region showing clearly nanocontacts formation. (c) Evolution time of the bridge structure.



(b)

FIG. 4. (a) SEM image showing another kind of membrane bridge between the macroscopic copper wires. Notice the presence of a nanocontact of  $\approx 70$  nm width (see arrow). (b) Chemical analysis performed on this nanocontact with an ultrathin window energy x-ray spectrometer showing a large copper signal but not any other contaminants as oxygen, carbon, etc.

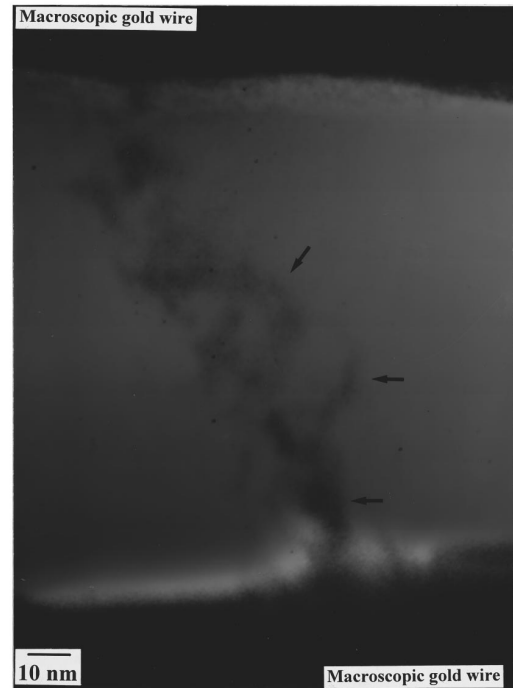


FIG. 5. TEM image showing a nanocontact with a filament structure between two macroscopic gold wires. The bridge is composed of nanowires less than 5 nm wide (see arrows).

macroscopic wires with a nanocontact of  $\approx 70$  nm width [see arrow in Fig. 4(a)]. The chemical analysis performed on this “bridge” with an ultrathin window energy x-ray spectrometer shows a large signal of copper [Fig. 4(b)] but not any other contaminants as oxygen, carbon, etc. The probe size used during microanalysis was  $\approx 200$  nm for copper detection.

Moreover, in order to increase the size resolution in our experiments, a TEM study has been performed using a similar experimental setup. A nanocontact with a filament structure has been revealed by TEM between two macroscopic gold wires (see Fig. 5). This “bridge” is composed of nanowires less than 5 nm wide (see arrows in Fig. 5).

These experiments have been performed with different metals and the nanocontacts have been observed with copper and gold macroscopic wires. However, this phenomenon did not occur with tungsten, which presents a more rigid structure. Thus, the nanocontact formation could depend on the plastic properties of the wires used and this experiment could give information on the plasticity level of the studied material.

In conclusion, we have depicted in this paper experiments in which the structure of contacts breaking in time between two macroscopic metallic (gold or copper) wires has been observed by transmission and scanning electron microscopy. These experiments show that the contact breaking is not done in a clean way but stretched into nanofilaments. Moreover, it should be composed of several stages, the last one leading to nanowire formation. The SEM and TEM images displayed in this paper correspond to different stages of the contact breaking. The chemical nature of these “bridges”

has been checked by electron dispersive x-ray spectrometry revealing only a copper or gold signal depending on the macroscopic wire used. The nanowires observed by TEM during the last stage of the contact breaking should lead to CQ as reported in early experiments and theory.<sup>1-8</sup>

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