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Low temperature (LN_2) and UHV mechanically controllable break junction setup to study quantum electrical transport of atomic-size metal nanowire

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Abstract. Reliable metal nanowire studies requires experimental stringent conditions, as clean samples and environment. In this sense, we have designed and built a dedicated instrument to study electrical transport properties of atomic-size metal contacts based on the mechanically controlled break junction technique, operating at ultra-high-vacuum conditions. Here we describe the chosen setup, its implementation and performance.

1. Introduction

The development of instruments where it is possible to study, manipulate and characterize nanometric objects have opened a wide variety of possibilities for basic science and technological purposes. In particular, it has been possible to study the electric transport through atomic size metal wires (NWs). In these systems the conductance is quantized, following the relation $G = nG_0$, where n is an integer, $G_0 = 2e^2/h$ ($\sim 1/(12.9k\Omega)$), e is the electron charge and h is the Plank's constant [1]. NWs can be generated in a simple way: touching two metal surfaces and subsequently pulling them apart; due to wetting, a nanometric bridge forms between the surfaces and, just before breaking, the contact is only composed of a few atoms [2]. However, this kind of procedure renders difficult the experiment interpretation because a new NW is generated for each conductance acquisition and its atomic arrangement follows different paths during the elongation [3]. In consequence, the conductance plotted as a function of elongation (hereafter named conductance curves) show different profiles, although all curves show conductance plateaus separated by abrupt jumps [2, 4, 5]. The procedure used to overcome this difficulty is a simple statistical analisys of an ensemble of conductance curves. Instead of considering the conductance as a function of the elongation parameter, the electrical transport can be represented as a histogram of conductance ocurrence. Here a flat plateau generates a histogram peak. By adding the histograms from a ensemble of measurements, a global histogram (GH) is obtained, which represents the general tendency of the transport behaviour. The GH usually shows well defined peaks close to the integer multiples of G_0, fact that can be considered the proof of the conductane quantization in NWs [2, 4]. It must be remarked that we are dealing with simultaneous changes of atomic and electronic structure, which should be discriminated in order to obtain a precise interpretation of the conductance curves [3, 6–8]. However this statistical procedure hides all

NW structural information. Also, as NWs are generated by mechanical deformatin, their atomic arrangement evolution should be influenced by the formation and moviment of structural defects, properties that change strongly temperature [9].

The experiment that has been mainly used to study atomic size NWs is the Mechanically Controllable Break Junction (MCBJ) approach [2, 10–13]. In a MCBJ, a metal wire is two-point fixed on a flexible substrate (plastic or isolated CuBe); subsequently a linearly moving tip is used to bend the substrate and break the wire in a pre-fragilised position (Fig. 1). Thereafter NWs are obtained touching/separating the two fresh surfaces by bending the substrate with fine linear tip movements. One advantage of this system is that the tip movement is not transferred to the NW directly, but through the bending of an elastic substrate, which acts as an effective reduction of the linear movement and give more mechanical rigidity to the system. Moreover, the NW can be generated from fresh surfaces produced by the wire rupture, process that can be performed *in situ* in a vacuum chamber. This aspect becomes crucial because a NW showing quantized conductance (QC) must be composed of one/few atoms and any contamination may induce a drastic change in the NW properties [3, 14–19]. In fact, for a pressure in the 10^{-4} Pa range, one monolayer of the chamber residual gas is formed on a sample surface in ~ 1 second, while it takes some hours for pressures in the ultra-high-vacuum (UHV) range ($< 10^{-8}$ Pa). Usually, a conductance experiment involves the acquisition of many curves (hundreds), each one lasting in the millisecond to second range. Then, UHV conditions will be required to fulfill the stringent cleanness needs.

We use a time resolved high resolution transmission electron microscope (JEM 3010 URP, 1.7 Åpoint resolution, LME/LNLS, Campinas, Brazil) to study the NW structure. This instrument enables us to follow the formation/evolution of NWs at room and liquid nitrogen (LN_2) temperature. Thus, in order to correlate the atomic arrangement and the conductance of NWs (the correlation procedures are described elsewhere [3, 6–9, 20]) the MCBJ must be capable of study electrical transport at these two temperatures values [9].

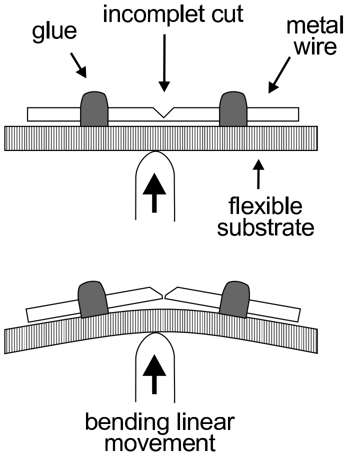

Figure 1. Operation schema of the MCBJ. The substrate is bent to break the wire sample in the pre-fragilized position. Then, the separated parts are put together again in a controlled way in order to generate NWs.

Here we describe the design and construction of a temperature controlled UHV compatible MCBJ dedicated to metal NW conductance studies. We present an overview of the developed experimental facility, its operation, control and performance. The cleanness and purity of the generated NWs open new perspectives for the study of these nanometric systems both from the reliability of the conductance measurements, as well as from wide range of materials that can be analyzed in the UHV environment.

2. Instrumental Details

2.1. Vacuum System

The instrument chambers were built using materials that are compatible with UHV and also avoiding the generation of small cavities, which may limit the attained final pressure because they act as internal leaks. In addition, the chamber was designed with a geometry that maximizes gas conductance and pumping rate. The whole MCBJ mechanism and the associated electrical feedthroughs are mounted on a single flange (CF200), which is positioned at the top of the UHV chamber. This configuration allows fixing the complete experimental apparatus in a single block, avoiding connections through the UHV chamber and, in addition, the MCBJ setup can be easily removed for sample exchange and maintenance. The primary pumping set is composed of an oil free mechanical and a turbo drag molecular pumps, which cover the atmosphere to $\sim 10^{-4}\ Pa$ pressure range. In order to reach pressures $< 10^{-8}\ Pa$, we have employed a 150 l/s ion pump and a home-made LN_2 cooled titanium sublimator. They are mounted at the bottom of the whole system in order to reduce the ion pump magnetic field and also to prevent contamination from the titanium evaporated inside the sublimation system.

2.2. Break Contact System

As mentioned previously, a break contact system is conceptually very simple, as it only contains one mobile part composed of a linearly moving tip, which deforms a flexible substrate. The sample holder is basically a fixed part, which supports the substrate through two points against which it is pressed and deformed by the tip. We must keep in mind that the substrate also acts as a reduction mechanism for the NW deformation of about a factor of 100.

The linear movement fulfills two distinct functions, what requires two different operating regimes. The first one concerns the substrate bending with a rough movement that is initially used to break the wire in UHV conditions; subsequently a more precise movement is required to generate the NWs in a controlled way by approximating/separating the wire parts. The coarse movement is performed by a home-made linear feedthrough that uses a screw to convert rotational movement, that is drived by a 3200 step motor, into translation. It was built to have a displacement increment of 0.15 μm and total range of 10 mm ($\sim$ 66000 steps).

A piezo actuator located at the extreme of the moving tip performs the fine movement. We have used a commercial multilayer piezo actuator (Morgan Matroc 70037-2) in order to have both a precision tip displacement (10 nm/V) but also with a reasonable total range (3 μm) at low applied voltage (300 V). In this geometry all elements (coarse and fine movements) are lined up so that the fabrication and subsequent assembly turns out to be rather easy (Fig. 2).

In order to render easy the sample exchange, we have designed a separated and extractable block (a cassette) where the sample is mounted. This cassette allows an easy and quick fixation and adjustment of the substrate in the final position outside of the instrument and without any manipulation of the MCBJ head and tip. The sample preparation procedure includes a series of steps that must be carefully performed, and are described in detail in this article.

2.3. Cold Finger

As explained above, NWs should be generated and measured at room and LN_2 temperatures. We use a LN_2 cold finger, composed of a LN_2 reservoir coupled to a copper rod, installed in the same CF200 that holds the MCBJ system (Fig. 2). The sample is cooled by three cooper strands that connect the cold finger to the two point support of the flexible substrate and the bender tip. To avoid that the sample acts as a vacuum pump due to the low temperature, contaminating itself, one cage rigidly connected to the finger protects the MCBJ system. In a first moment, the finger and the cage is cooled down while a current is applied to the sample. This procedure guarantees that residual gas surrounding the sample is trapped and, in consequency, we have a lower pressure in this region. Next the sample is cooled by turnning off the current and

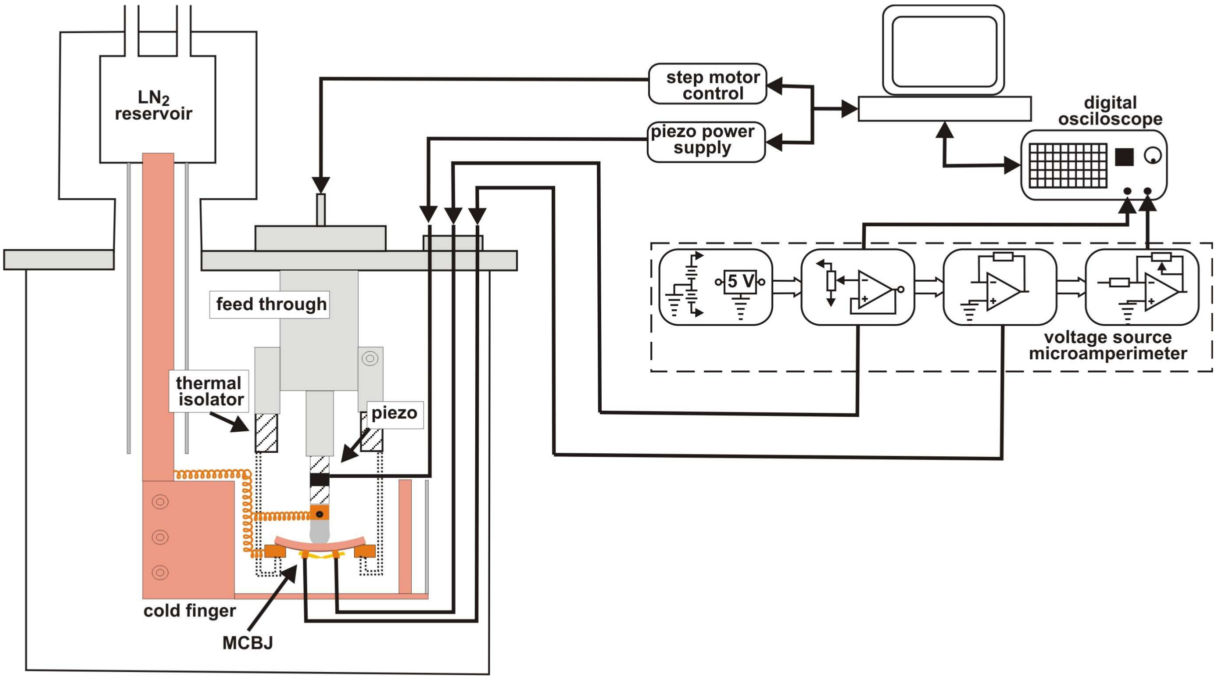

Figure 2. MCBJ schema showing the cold finger and a diagram of the control and data acquision systems

the experiment can be done. The temperature is controlled by two thermocouples, one on the flexible substrate of the sample holder and the other on the cold finger.

2.4. Sample Preparation

The sample preparation requires several steps in order to solve different important aspects: a) fixation of the wire on the substrate; b) electrical contacts; c) fragilization of a particular wire position and d) installation in the MCBJ.

We have used CuBe as flexible substrate, but also other materials such as plexiglas could be chosen. To two point fix the sample, our experiments showed that the best fixation mechanism was the use of metal clamps isolated from the substrate. This system has showed advantage because we can control the distance between them and it show good electrical contacts; subsequently, the sample wire may be easily pre-fragilized by a incomplete knife cut. However this procedure generates important residual stresses in hard materials such as Ni, what may induce that the tips stay separated after the initial rupture, invalidating the experiment. In order to minimize this effect, we perform the cut in a slightly pre-bent substrate already installed in the cassette. This procedure guarantees a minimal sample manipulation after the wire fragilization, and also a quick installation in the MCBJ. Once the cassette is located in the instrument, only the two electrical connections from the sample to feedthrough remain to be made.

In order to clean the sample after its installation in the vacuum chamber, an electrical current is applied to the sample wire when the vacuum reaches $\sim 10^{-8}$ Pa. This procedure rises the vacuum pressure, indicating that the sample temperature is increased and sample contamination is released. The complete wire rupture is performed only when the vacuum pressure recovers values $< 10^{-8}$ Pa.

2.5. Measurement and Control Electronics

To measure the NW electrical properties, we have developed an electronic system consisting of a home-made voltage source and current-voltage converter (Fig. 2). Both the NW applied voltage and the flowing current (converted to voltage) are acquired by a digital oscilloscope (Tektronic TDS540C). In this way, the conductance behavior is obtained *a posteriori* by the ratio of the two digitized measurements. The system has been designed to be as simple and fast as possible, where only strictly necessary functions were implemented. We have tried to minimize all possible sources of noise, for example the voltage source and current-voltage converter are powered by an insulated battery, while other electronics (oscilloscope, piezo power supply, etc.) are powered from the electrical network through a voltage transformer. Also, the whole system was screened against electromagnetic noise and the wires was arranged to avoid ground loops.

This electronics allows conductance measurement with an absolute error of 10^{-4} G_0, derived by using several calibrated fixed resistance in the 1 to 50 kΩ range. This precision represents an important progress in the NW field because reported experiments usually show error about two orders of magnitude larger ($\sim \Delta G/G < 10^{-2}$)[5].

Most of metal NWs studies are based on statistical analysis that yield average behaviors of hundreds or thousands of conductance curves [2]. This fact points out the need of a system that should generate NWs and measure their conductance behavior with the maximum efficiency, if possible with a high degree of automatization. In the MCBJ described here, the whole bender mechanism and the data acquisition are fully software controlled. This capacity allows us to record automatically series of conductance curves obtained in identical experimental conditions.

3. Discussion

The performance of the developed experimental setup can be illustrated by the curves of conductance vs. time showed in Fig. 3 for Au NW at LN_2, room temperature and applied voltage of 100 mV (current $\sim 7.7\mu A$ for $G = G_0$). These curves are examples extracted from series of 1000 curves that have been automatically acquired after breaking a 75 μm in diameter Au (99.99%) wire in a vacuum of $< 10^{-8}$ Pa. They display remarkable clean flat plateaus separated by abrupt jumps, indicating clearly the discrete nature of the NW conductance.

By carefully analyzing the slopes of the jumps and the flatness of the plateaus, we can derive important information on the performance (speed and noise level) of the electronic measurement system. The jump between discrete levels (distance $\sim 1\ G_0$) shows extremely small curvature and in average it has been measured to occur in the $\sim 10\ ns$ range, demonstrating the fast response of the current-voltage converter. If we assume that a flat plateau represents a conductance with identical value during a certain period of time, the dispersion of conductance values will give an indication of the electronic noise during the experiment. For a extremely flat conductance plateau at $\sim 1\ G_0$, a dispersion of $\sim 0.005\ G_0$ are usually obtained, what is surprisingly 2 order of magnitude higher than when measuring calibrated conductance setups in the same configuration [2]. This fact suggests that this noise level could be partially associated to the intrinsic properties of electrical transport in NWs and work is in progress to elucidate this point.

As explained above, all conductance curves show different profile, so that most NW studies rely on the analysis of average behaviors of many curves. Fig. 4 shows GHs for series of 1000 measurements at room and LN_2 temperature. The presence of peaks close to integer multiples of G_0 has been considered as the proof of the QC in atomic-size NWs [2, 4, 21].

Concerning the room temperature experiments, the general aspect of the curves in Fig. 3, measured in UHV, is quite similar to analogous data obtained in poor vacuum or atmosphere conditions [4]. An analogous conclusion can be deduced when looking at the GH in Fig. 4, where the peaks are dislocated to the left side of integers multiple of G_0 [2, 4, 21]. However this rather simplistic analysis may led to the erroneous evaluation that UHV conditions may not be required for the study NWs. Studies of NW conductance have revealed that crucial

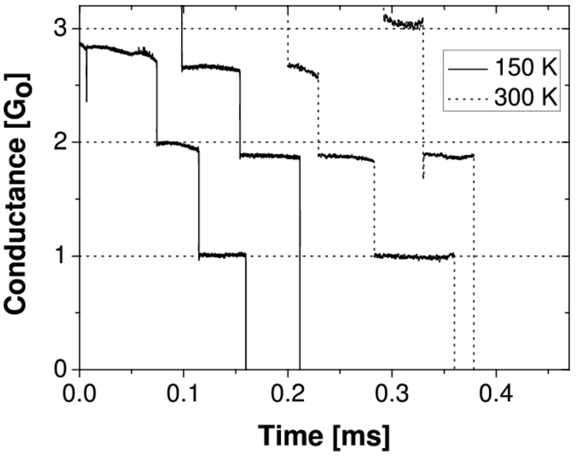

Figure 3. Au NW conductance obtained from series of measurements performed at room and LN_2 temperature. It must be observed that all curves have different profiles, although they show clear plateaus near integer multiples of G_0 and separated by abrupt jumps.

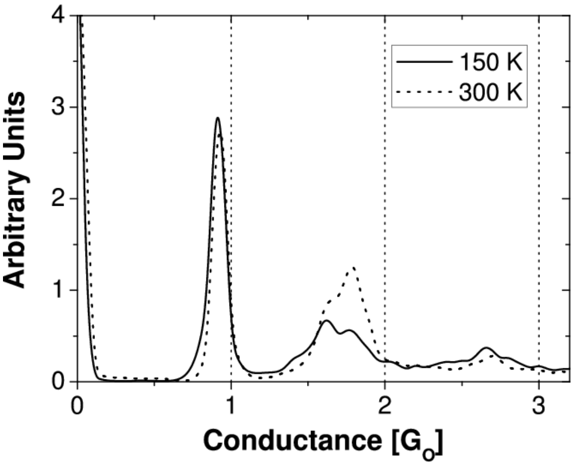

Figure 4. Global histograms of Au NWs measured using the UHV-MCBJ at room and LN_2 temperature. A set of 1000 conductance curves were used for each histogram.

differences are observed in the voltage dependence (non-linearity) between Au NWs generated from contaminated and clean surfaces [14].

4. Summary

The experimental study of quantum effects in the electrical transport of atomic size wires presents challenges concerning the control of atomic arrangement generation and evolution, the precise conductance plateau measurement and the contamination control of the studied NW.

Our system allows the study of clean metal NWs because the surfaces used to generate the contacts are obtained in a pressure $< 10^{-8}$ Pa. This renders possible study the conductance of reactive materials, as metal that present magnetic interest (Ni, Fe and Co [20]). Other possibility concerns the study of the valence number role on the NW conductance behavior. In addition, the obtained precision on the conductance measurement allow performing a precise measurement of the deviation of the expected quantized value. Finally, temperature dependent experiment are essential to generate a unified interpretation in the field of quantum conductance of atomic size NWs generated by mechanical deformation.

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