INTDS NEWSLETTER

Contents

<u>Cover picture</u>:.
Control terminal of the 150 MeV Geel Linear Accelerator (GELINA).

Editor's note

Enclosed separately with this newsletter is a ballot paper with voting instructions for the 2002 election for the INTDS board. Please return your ballot paper to Hans Maier, chairman of the election committee before October 25, or hand it in at the conference in Argonne. Only <u>INTDS members</u> may vote.

Please also note the conference annoucement for the INTDS conference in Argonne, 4-8 November. The registration deadline is 31 July 2002.

Chris Ingelbrecht

Editor

21st World Conference of the INTDS

Accelerator Target Technology for the 21st Century

November 4-8, 2002 Argonne, IL USA

Argonne National Laboratory

The conference will cover classical and innovative target preparation related to the topics below.

- Development of targets, thin films and foils used in studies conducted with low and high-energy accelerators and research reactors.
- Foils used in space science for imaging of solar system and galactic neutrals, and those used for space debris, erosion and penetration studies, etc.
- Synchrotron targets, e.g. used to quantify beam polarization or for beam-intensity profiles.
- Chemical and mechanical processing of targets and foils for medical isotopes and enrichment.
- Mechanisms for producing secondary beams, including gas targets and catcher cells.
- Performance and characterization of targets used in nuclear physics experiments, transition-radiation production and heavy element synthesis.
- Design of high power targets and ion sources for radioactive beam production.

Publication of Proceedings: Nuclear Instruments and Methods

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Superconducting- Like Behaviour in Carbon Arc Films

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We present the experimental results obtained with the help of such techniques as magnetic force microscope (MFM), dc SQUID magnetization, reversed Josephson effect, and resistance measurement in carbon arc films. From the dc SQUID magnetization measurement, the size of elementary superconducting loop is 102 nm and it is also found in MFM images. The observation of induced voltage and its rf frequency, input amplitude, and temperature dependence reveals the existence of Josephson Junction array. All the data related with the existence of high temperature superconducting-like phase or fluctuations up to 650 K.

I. INTRODUCTION

Recently superconducting-like behaviour was found in highly oriented pyrolitic graphite (HOPG) [1-3]. Also some weak superconducting behaviour has been observed in carbon films formed by sputtering of spectral pure graphite in an electrical arc discharge (CA films) [4-5]. The essential of these two materials is that there is some paramagnetic response under magnetization measurements at high enough magnetic field. The explanation of such kind of behaviour in graphite due to ferromagnetic impurity influence is questionable for many reasons [1]. As for the CA films, the absence of unpaired spins and paramagnetic signal has been proved early in ESR experiment [4]. On the other hand the existence of superconducting-like hysteresis loops in HOPG [1], reversed Josephson effect (RJE) in CA films [4] and also the superconductivity in graphite-sulphur mixture at *35 K* [6] give us some idea of coexistence of superconductor (SC) and paramagnetism (PM) in pointed materials. This is not so surprising neighbouring in high-temperature SC when cooled through their transition temperature in a small external magnetic field. This result known as the paramagnetic Meissner effect (PME), contrasts with the standard diamagnetic response of classical superconductors and has been the subject of extensive investigations for the last years. An experimental study of the paramagnetic response in Bi-based polycrystalline SC was first made by Braunisch *et al*.[7] and subsequently reported in many other high-temperature superconductor (HTSC) [8-11].

Some theoretical work suggested that PME might be attributed to the magnetic impurity between the grains forming the anomalous Josephson junctions (π-junctions)[12] or non-*s*- wave pairing symmetry [13]. Also the topological disorder enhances the density of state in graphene sheets and can give rise to coexistence of SC and PM fluctuation [14].

Common feature of all systems pointing above is their granular structure forming the Josephson-junction arrays (JJA). Therefore a lot of modelling experiments have been made to check the existence of PME in JJA. Dominguez *et al*.[15] have modelled a granular SC as a mixture of conventional and π-junctions network where the nodes represent the grains and the links represent the coupling between them. In this case, the low-temperature and zero-field-

cooling (ZFC) magnetic susceptibility was of the order of *-1* (in SI units), while the field-cooling (FC) susceptibility was paramagnetic for some small value of magnetic field. Other models based on networks of conventional junctions could explain the PME [16-19]. In the experiments [18] the JJA of conventional junctions has been used with the elementary loop of size of *46 μm* consisting of four Josephson junctions. In the *ac* susceptibility measurements up to the magnetic field h_{ac} of about *50 mOe* which corresponds to *5Φ₀* per elementary loop the diamagnetic signal has been observed. At the increasing of h_{ac} above *50 mOe* the PME has been observed as the re-entrant with temperature positive magnetisation. As it has been shown this no monotonic behaviour of magnetisation is the cosequence of the magnetic field dependence of critical current in elementary loop (Fraunhofer pattern).

The difference situation in CA films and HOPG materials from that of high-temperature SC and their JJA model is in the scale of magnetic field. In our case the magnetic field where the PME occurs is about *1T* as compared with the *50 mOe* in the experiments of Refs. [18-19]. However the MFM study reveals the size of elementary conductive loop of granules in CA films being about *500* times smaller than in HTSC giving rise to the same magnetic flux per SC loop. So it is believed we can proceed our measurements and analysis partly in a fashion similar to Refs.[16-19].

This paper presents the results of measurements of *dc* magnetisation, magnetic force microscope, low and high temperature electrical resistance, and RJE voltage of CA films. Each of this results taking alone from others can be explained ambiguously but been linked together they gives the evidence of existing of SC phase or fluctuation in CA films.

II. EXPERIMENTS

The CA films for our measurements were prepared by arc evaporation of *99,999%* purity carbon source material onto quartz substrate at room temperature. The potassium-oleate ($C_{18}H_{33}O_2K$) has been used as a release agent at subsequent separation of self supported film from the substrate under the floating procedure in distillate water. The CA films were annealed at *1000 C* for *10* hours and the thickness of annealed films is about *960 nm*. The *2 MeV H$^+$*-PIXE analysis indicate that Fe concentration in the annealed films is *185 ± 38 ppm* and film density is about *2.25 g/cm^2*.

The temperature dependence of resistance was performed in a commercial closed cycle refrigerator and in a vacuum furnace, below and above room temperature, respectively. The temperature range of closed cycle is *20 K< T <325 K* and 300 K < T < 777 K for vacuum furnace. In low temperature resistance measurement, a Lakeshore of *330* temperature controller digitally records the sample temperature using a RS-Components of *PT-100* platinum resistor placed directly on the sample. With this cryogenic system, samples can be cooled to *20 K* in about *2 h*. Warming up of the system takes longer, up to *10 h* to go from the lowest temperature to room temperature. A *Keithley 220* programmable current source is used to supply a current *I*, a value that is also monitored by a four-wire precision resistor of *0. 1Ω*. The voltage drop *V* on this resistor and on the sample is measured with a *Keithley 2182* Nanovoltmeter. The *Agilent 34970A* Switch Unit, with a *34901A 20* Channel Multiplexer card and a *34970A 4×8* Matrix Switch card are used to allow for switching of both voltages and the current polarity respectively, interconnecting the test leads with the nanovoltmeter and the current source in a break-before-make mode. All these instruments have *IEEE-488.2* interfaces and use an *IBM*-compatible personal computer as the controller. The control software, written in LabVIEW from National Instruments, programs a current value and selects the current polarity from the source to the sample *I*. Therefore both the drop of voltage *V*, the sample temperature *T*, and the time *t* are

digitally recorded. All measurements are continuously stored in *ASCII*-files while the sample is either cooled down or warmed up.

To provide the four-probe electrical resistance measurements the thin gold wire electrical contacts has been attached the film samples with the help of silver paste. Usually contacts have dimensions of *3* millimetres long and *1* mm width.

MFM measurements have been employed to study the topography of film surface and possible magnetic clusters. Magnetic force gradient images and sample topography were obtained simultaneously with a *Nanoscope III* scanning probe microscope from Digital Instruments. The microscope was operated in the "*tapping/lift* ^{TM}" scanning mode, to separate short-range topographic effects from long-range magnetic signal. The scanning probes were batch fabricated *Si* cantilevers with pyramidal tips coated with a magnetic *CoCr* film alloy. Prior to acquiring image, the probe was exposed to a *3 kOe* magnet which aligned its magnetization normal to the sample surface direction (i.e., *z* direction). All MFM data shown in this paper were collected with tip magnetized nearly perpendicular to the sample surface, making the MFM sensitive to the second derivative of the *z* component of the sample stray field. To exclude any influence of the MFM tip on the sample micromagnetic structure and to verity that any non-*z* components of the tip magnetization contributed negligibly to the MFM measurements, images were taken with various tip-sample separations (*10 ~ 100 nm*) and in this measurement, the general shape of MFM images is not change with the tip-sample distance.

III. RESULTS AND DISCUSSION

In order to confirm the existence of JJA in our carbon ark films, we have performed the measurements of RJE. We have studied the RJE in various experimental arrangements like that in Ref. 23 and 24 and measured induced *dc* voltage, V_{dc} as a function of frequency, temperature, and input *ac* amplitude. The fluctuations of induced V_{dc} are fast and it may be related with a series combination of a large number of individual Josephson junctions. The dependence of induced V_{dc} on the *rf* frequency at room temperature is shown in Fig. 1. In this measurement, we used the same circuit as in Ref. 23. Note that there is the polarity reversibility of V_{dc} which is one of the tests which can distinguish the RJE from the commonly known rectification effect [23]. Moreover, in our CA films, there is a hysteresis in induced V_{dc} with frequency as shown in figure 1, i.e. V_{dc} depends on the direction of frequency change for the frequency f > 9 MHz. This hysteresis has not been found when we used the circuit of Ref. 24.

Figure 2 shows the change of induced V_{dc} with the input *ac* amplitude, V_{ac} for several fixed *rf* frequencies by using the same circuit as in Ref. 24. For a small *rf* frequency (f < 1.6 MHz) V_{dc} increases with *ac* amplitude and changes the sign at f = 1.6 MHz (see the inset of Fig. 2). For small V_{ac} it is difficult to find the change of polarity, however, for higher V_{ac} it is possible to resolve the polarity change of induced V_{dc} with *rf* frequency.

For comparison, we use standard carbon resistor, whose resistance is the same as a carbon film and measured by means of the same manner. There is *NO* change of polarity in standard carbon resistor and the amplitude of induced V_{dc} is one order of magnitude smaller than that of carbon arc films. This reveals that polarity change of induced V_{dc} is a peculiar signal of carbon arc films.

The temperature dependence of induced V_{dc} was measured by using the electrical circuit of Ref. 24 at fixed input amplitude and *rf* frequency. At all temperatures, the data were obtained after the thermal equilibrium. The result is shown in Fig. 3 for 300 K < T < 777 K at V_{ac} = 10 V and *rf* frequency = 1 MHz. The induced V_{dc} exponentially decreases with temperature and this can be successfully fitted by the form $V_{dc}=\alpha e^{bT}$ for 300 K < T < 650 K, where *1/b= -104±60 (K)*

is in good agreement with the result of Refs. 4, 5, and 24. Around 650 K it goes to zero and have minus values above 650 K. Above 650 K, the change of induced V_{dc} with temperature does not follow the exponential behaviour. From 300 K to 777 K, the change of induced V_{dc} is more than 100 % and it is larger than the change of resistivity (17 ~ 25 %) in the same temperature range.

In fresh films there has been the evidence of corrugating and warping of surface in topographic image and the absence of magnetic clusters or domains in MFM images (see Fig. 4). Here scan size was 5 μm x 5 μm and the scan height in MFM measurement was 50 nm. The annealed films have a smooth surface and revealed the topographic clusters with the average size of about *165 nm* (see Fig. 5). As you can see the MFM image in figure 5, it is possible to resolve the magnetic domains or particles. After magnetization of films with the use of permanent magnet with the magnetic field of *H =1kOe* there has been clear evidence of magnetic clusters with the same size as the topographic clusters. This reveals that there are the magnetic clusters or domains in annealed CA films and the magnetization of magnetic domain is small, which however can be resolved in SQUID measurements.

DC magnetization *M(H,T)* measurements were performed with the SQUID magnetometer MPMS7 from Quantum Design. The *M(H)* curves for different temperatures are shown in the Fig. 6. As can be seen from Fig.6 there is some paramagnetic response at *H<10000 Oe*. At *H>10000 Oe* the signal is negative due to natural diamagnetism of graphite granules embedded in the matrix of both twofold and fourfold coordinated atoms [20-21]. After subtraction this linear diamagnetic background $M= -\chi(T)H$, we found the oscillating behaviour of magnetisation (see inset of Fig. 6) which is similar to the dependence of the Josephson junction critical current on a magnetic field applied in the plane of the junction. This results is in agreement with the ones obtained in Refs.[17,18,22] with only one difference in the scale of magnetic field. As it is well known the minimums of critical current in the Fraunhofer pattern correspond to integer magnetic flux quantum. Therefore for the first minimum in correspondence with Refs.[18-19] we have the relation:

$$5\Phi_0=\mu_0 a^2 H , \qquad (1)$$

where Φ_0 is the magnetic flux quantum, μ_0 is the vacuum permeability, *a* is the size of elementary SC loop of granules which are in the state of common phase coherence. This means that inside this elementary loop of granules the SC current can flow. The first maximum of *M(H)* in Fig. 6 (which correspond to first minimum of critical current in Fraunhofer pattern) gives the value of *a* about *102 nm* as compared with the *46 μm* in the experiments described in Refs.[18-19]. This produces the magnetic field scale *2x10⁵* times larger in our case.

The data of Fig.6 also provide us with the value of critical current I_c of elementary Josephson junction composes the film granular structure. For this purpose we can use the relation from Ref.[18] which connect the magnetisation with I_c:

$$M = \frac{LI_c}{\mu_0 a^2} \qquad (2)$$

where *L* is the inductance of the elementary loop. This gives us the value of *I_c(100K)~0.87 μA* for the annealed film. This value gives the upper evaluation of the critical current density of about *10^4A/cm²*. However taking into account the cross section of current flow between adjacent loops must be substantially smaller than the loop square we can obtain the current density close to pairs breaking value. It is interesting to note that the critical current in non-annealed film is evaluated

to be *30* times larger than in annealed one. This means that the annealing may give rise to suppress the Josephson pairing between the granules. In this sense the observed corrugatness and warpness of the unannealed film surface may turns to be the same topological disorder which enhances the density of state at the Fermi level of graphite sheets as it is shown in the Ref.[14].

The size of elementary loop inferred from the magnetisation experiments is nearly the same as one observed in MFM –measurements (see Fig.5). So it is believed this coincidence is not a casual but is the evidence of existence of SC loops in the CA film samples.

III. SUMMARY

- Experimental results presented in this work has been obtained with the help of such techniques as *dc* SQUID magnetisation, MFM, RJE, IV, and *R(T)* measurements. This results support the existence of SC phase or fluctuation in CA films at room and possibly higher temperatures.
- From the *dc* magnetisation measurements the size of elementary SC loop of *102 nm* and the critical current in such loop of *0.87 µA* has been obtained. This value has been found to be in agreement with both the MFM.
- The observation of *dc* voltage induced in samples due to RJE and their frequency and temperature dependencies, which agree well with the previous observations, has been considered as independent proof of existence of JJA and therefore the presence of SC phase or fluctuation inside CA films.
- The disappearance or change of polarity of RJE signal above 650 K reveals that SC phase persists up to 650 K.

REFERENCES

1.   Y. Kopelevich, P. Esquinazi et al., J.Low. Temp. Phys. 119(2000)691.
2.   Y. Kopelevich et al., Fizika Tverdogo Tela 41(1999)2135.
3.   H. Kempa et al., Solid State Communications 115(2000)539.
4.   S. G. Lebedev and S. V. Topalov, Bulletin of Lebedev's Physical Institute N11-12(1994)14.
5.   S. G. Lebedev, Preprint INR RAS, N 1033/2000, Moscow, 2000 (in Russian).
6.   R. Ricardo da Silva…..?
7.   W. Braunisch et al., Phys. Rev. Lett. 68(1992)1908.
8.   B. Schliepe et al., Phys. Rev. B47(1993)8331.
9.   S. Riedling et al., Phys. Rev. B49(1994)13283.
10.   U. Onbasli et al., Phys. Status Solidi B194(1996)371.
11.   G. S. Okram et al., Condens. Matter 9(1997)L525.
12.   F.V. Kusmartsev, Phys.Rev. Lett. 69(1992)2268.
13.   H. Kanamura and M. S. Li, Phys. Rev. B54(1996)619.
14.   J. Gonzalez et al., Phys. Rev. B63(2001)134421.
15.   D. Dominguez et al., Phys. Rev. Lett. 72(1994)2773.
16.   C. Auletta et al., Phys. Rev. B51(1995)12844.
17.   Mahesh Chandran, Phys. Rev. B56(1997)6169.
18.   P. Barbara et al., Phys. Rev. B60(1999)7489.
19.   A. P. Nielsen et al., Phys. Rev. B62(2000)14380.
20.   L. Salamanca-Riba, Nucl. Instr. and Meth. B7/8(1985)487.

21.   C. Z. Wang et al., Phys. Rev. Lett., 70(1993)611.
22.   W. A. C. Passos et al., J. Appl. Phys. 87(2000)5555.
23.   J. T. Chen et al., Phys. Rev. Lett. 58(1987)1972.
24.   R. Munger and H. J. T. Smith, Phys. Rev. B44(1991)242.
25.   H. M. Jaeger et al., Phys. Rev. B34(1986)4920.
26.   B. G. Orr et al., Phys. Rev. B32(1985)7586.
27.   B. G. Orr et al., Phys. Rev. Lett. 53(1984)2046.
28.   T. Venkatesan et al., Nucl. Instr. And Meth. B1(1984)599.
29.   Y. Yeshurun et al., Rew. Mod. Phys. 68(1996)911.

Figure 1. Frequency dependence of Vdc in Carbon Arc Film
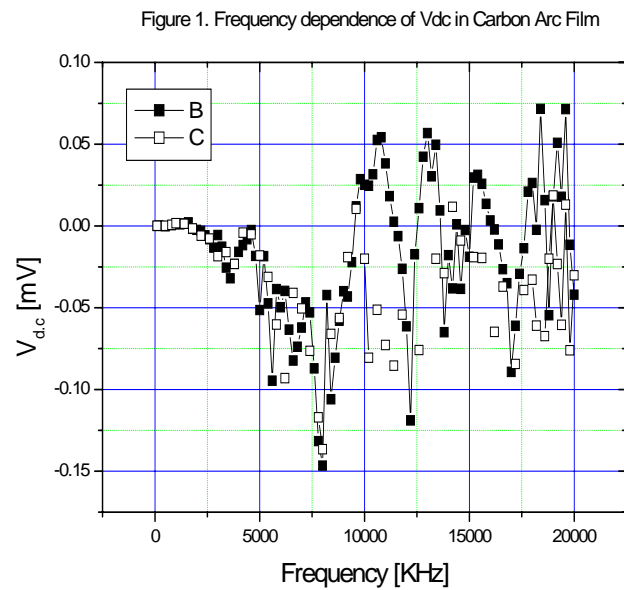
Figure 2. The dependence of the Vdc on the amplitude of rf signal at various frequencies.
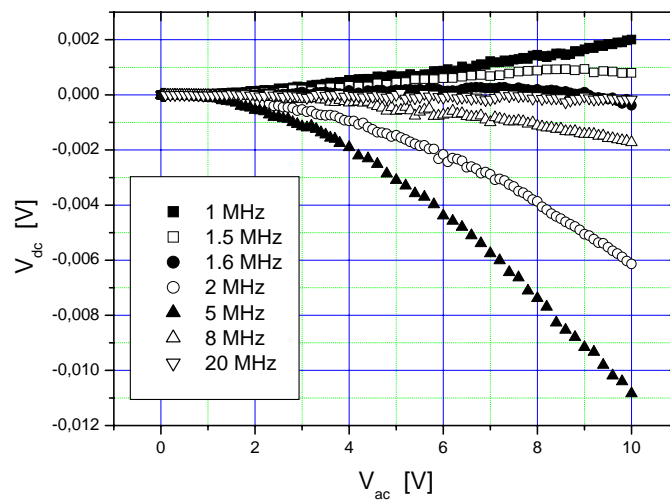
 Inset of Figure 2. Change of sign of Vdc under Vac variation.
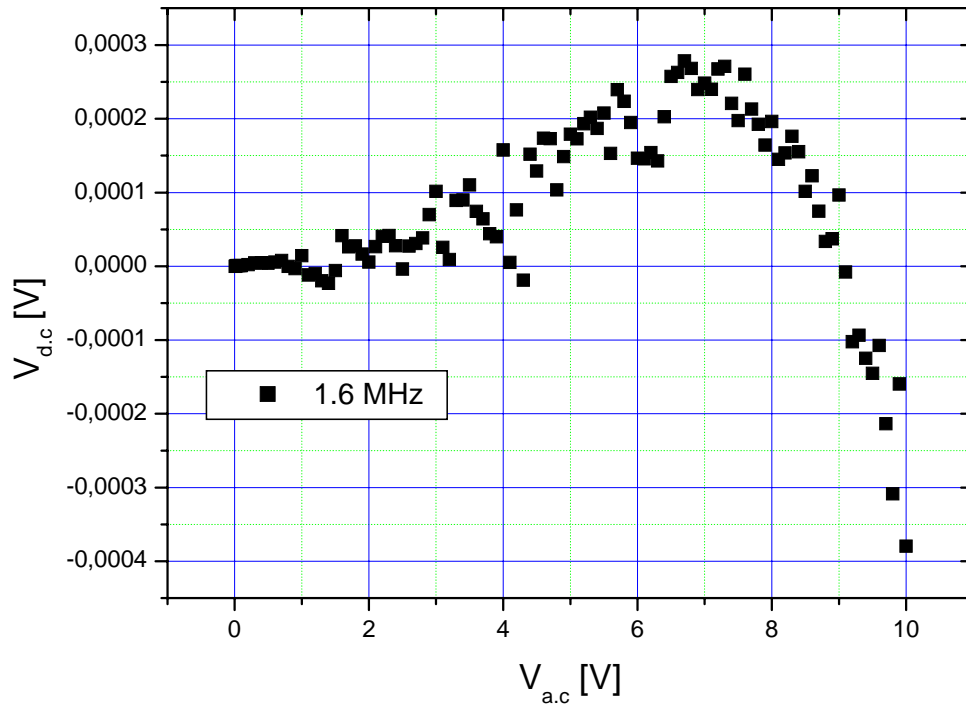
Figure 3. The dependence of Vdc on the temperature.
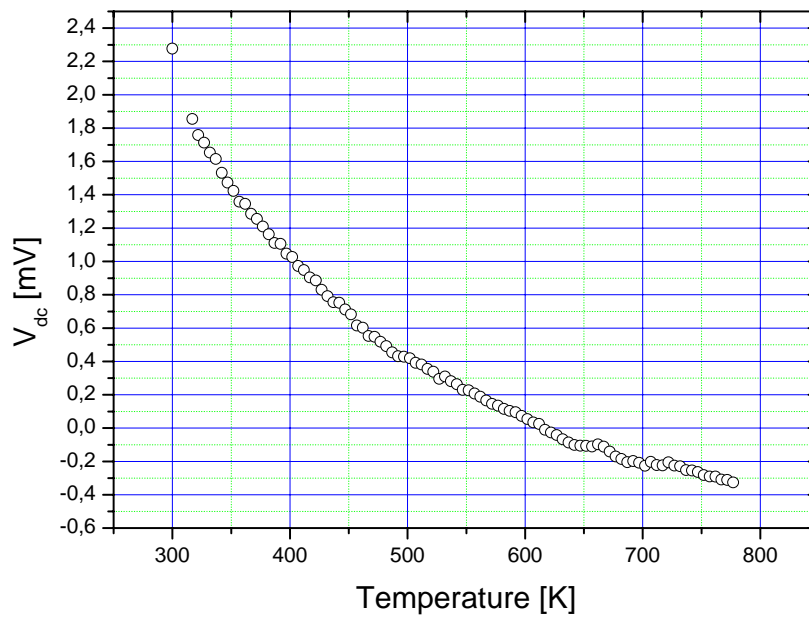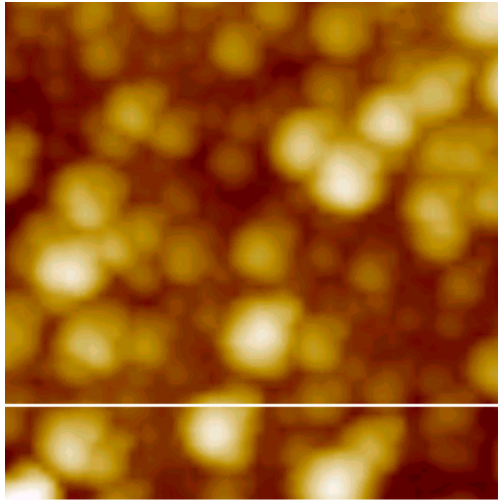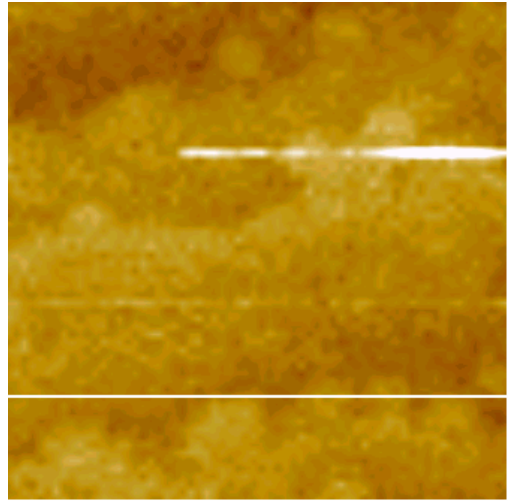
Figure 4. Topography (left) and magnetic force microscopy (right) pictures
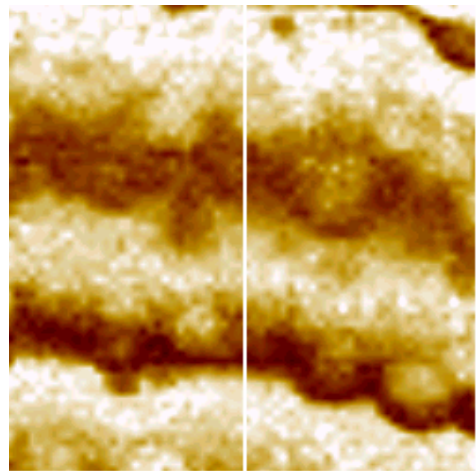
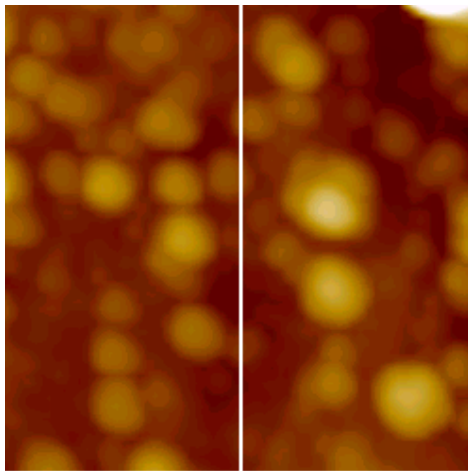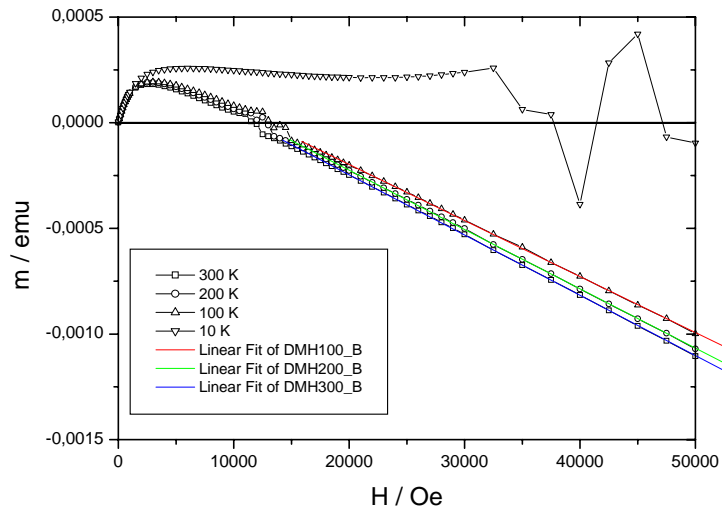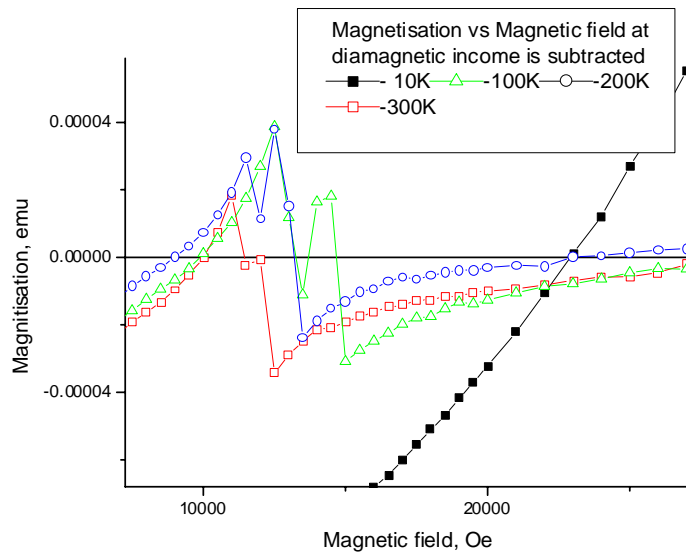

Topography

MFM image

Figure 5

Figure 6. SQUID magnitisation of CA film. The oscillation of magnitisation due to Josephson current can be seen.

Inset of Figure 6.

Preparation of [208]Pb targets with a rotaling substrate

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The recent experiment carried out at CIAE requested 24 targets of [208]Pb with the thicknesses of about 350µg/cm2.The target spot had to be on a Cu foil of 150µg/cm2 and should be 3 mm in diameter.

Considering the high cost of isotopically enriched material, rotating substrate holder[1] has been used for the preparation of the targets. Behrndt[2] expresses the film thickness d at a distance from the vertical axis of rotation by the equation.

$$d(s) = \frac{m}{\pi\rho} \cdot \frac{h^2\left(h^2 + r^2 + s^2\right)}{\left[\left(h^2 + r^2 + s^2\right)^2 - 4s^2r^2\right]^{3/2}} \qquad (1)$$
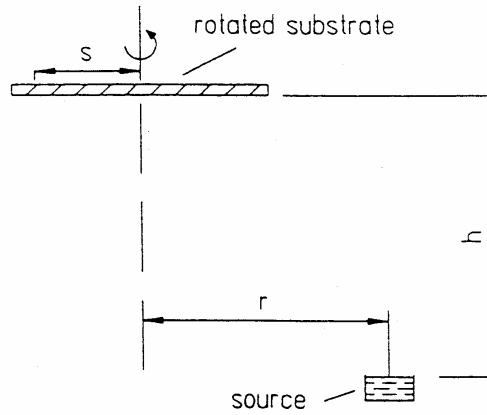

Fig. 1. Schematic drawing of the rotating substrate setup.

Using this formula film thickness distributions have been calculated and plotted in fig.2 for experimentally relevant values of h and r (fig.1). Eq.(1) was rearranged in order to obtain the target thickness units commonly used in nuclear physics.

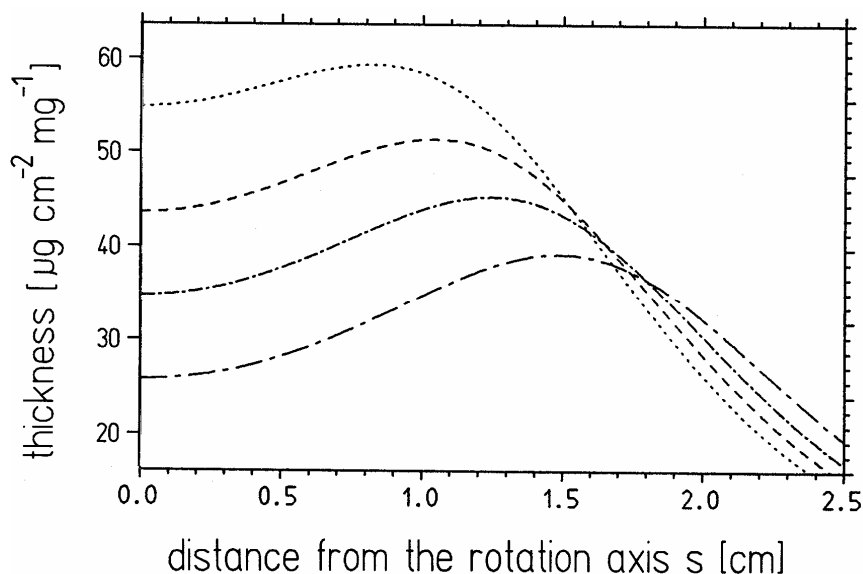$$d_n(s) = 1000 \cdot d(s) \cdot \rho / m[\mu g cm^{-2} mg^{-1}] \qquad (2)$$

fig. 2. Theoretical film thickness distributions for a source-to-substrate distance
h=1.3cm. Lowest dash-point curve is for a horizontal distance r=1.7 cm between source
center and substrate axis; upper dash-point curve is for r=1.5cm; dashed curve is for
r=1.35cm; pointed curve is for r=1.2cm

The distributions indicate that for a certain h there is a optimum r, which
results in both high collection efficiency and good uniformity of targets. The
optimum parameters for the spot target could be obtained from fig.2 (table 1).

Table 1 Optimum parameters

h/cm	r/cm	Collection efficiency/ μ gcm^{-2}mg^{-1}	S/mm
1.3	1.35	49.4	11-14

The substrate holder was stainless steel disk axially mounted on the shaft of
a small vacuum-qualified dc-motor. Four target frames with Cu backing of
150 μ g/cm^2 were mountes concentrically on the substrate disk. Ta boat of 4mm
wide and 20mm long was used as evaporation source. 7mg of ^{208}Pb on boat was
loaded for one evaporation and 4 targets were obtained. After six evaporation
runs 24 targets of 350 μ g/cm^2 were available using 42mg of ^{208}Pb.

References

[1]H.J.Maier.Proc.Workshop 1983 of the INTDS(Argonne Natl.Lab. ,ANL/PHY-84-2) p.109.
[2]K.H.Behrndt,Trans.Natl.Vacuum Symp.(American Vacuum Society,1963) p.379.

Experiences with platinum foils

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We needed Pt foils approximately 0.23 μm thick, corresponding to about 500 μg/cm2, mounted over 19-mm-diameter apertures in frames for a nuclear physics experiment. The frames were aluminum, 24 mm x 36 mm x 0.5 mm.

Evaporated platinum foils are typically made on heated substrates. The Movchan-Demchisin Zone II temperature limits (corresponding to 0.3-0.45 T_m, T_m = melting temperature) are 340-646 degrees C, which represents the approximate range of substrate temperature in which an evaporated compact nanocrystalline film is expected to be well consolidated without excessive tensile stress and without formation of large crystallites [1,2]. Pt foils are soft, with poor adhesion to metals [3]. Films only 35 nm thick on oxidized silicon are discontinuous, with cracks [4]. The minimum possible thickness for mounted foils appears to be about 40 nm (80 μg/sq.cm), according to Gursky and O'Rourke [5]. They deposited Pt foils on KI layers and found that the Pt layers were crystallographically aligned with the crystallites in the KI layers. Crystallite sizes were 300-1100 angstroms, for the thickness range used by Gursky and O'Rourke, and deposition temperature 280-350 degrees C, low enough (below Zone II) that the Pt layers perhaps were not well consolidated, but above the Zone II limits for KI (26-175 degrees C), so that those layers were certainly polycrystalline. Maier [6] made Pt foils down to 70 nm (150μg/sq.cm) by high-vacuum sputter deposition. Zell [7] made Pt on NaCl-coated glass down to 40 nm (80 μg/sq.cm).

Substrates were pieces of chromium-plated ferrotype plates [8] sheared to 114 mm x 114 mm lateral dimensions, washed with warm tap water and dish detergent, and rinsed dry. They were located about 20 cm above the electron-gun hearth. We considered using heated copper substrates without parting agent, then dissolving the copper to release the Pt films. However, Méens et al. [9] found that making 180-angstrom Pt foils on hot (300-degrees-C) copper substrates led to serious contamination of the Pt foils by Cu, due to the formation of the intermetallic $PtCu_2$ at the interface.

We used KI as the parting agent, with nominal thickness 200-300 nm, and substrate temperature 325-350 degrees C. This was evaporated from a closed kayak-style boat, resistively heated, offset about 10 cm from the electron gun. Gursky and O'Rourke [5] used KI, 110 nm thick. We found release to be poor using KI only 100 nm thick, perhaps because ferrotype plates are not as smooth as the glass substrates used by Gursky and O'Rourke. To get uniform thickness of the parting agent, we applied half of the desired KI thickness, then allowed the plate to cool to room temperature, opened the evaporator and rotated the substrate 180 degrees in its plane. Then the final half of the KI was applied and the Pt evaporated (both at elevated temperature).

We used a piece of Pt in a shallow, water-cooled copper hearth in an electron gun, directly below the substrate, as the evaporation source for Pt. Although the Inficon guide [10] claims that Pt can be evaporated from a tungsten filament, Maier-Komor [11] claims that Pt attacks tungsten, and we confirmed this. Zell [7] claims that Pt alloys with all metals, and evaporated Pt from resistively heated carbon rods. The compilation by Kurt J. Lesker Co. [12], and the review by Ross and Sontag [13], say that C crucibles can be used, and we have used them in the past effectively, but in the present case we wished to avoid as many sources of impurities as possible. We feared that some evaporation of a carbon crucible might occur if the electron beam struck it directly [cf. ref. 7].

As a test evaporation for Pt, we used 3.56 g of a previously-used globule of Pt (purity unknown) which was removed from a C crucible by hammering and then baked in air at 900 degrees C to remove residual attached carbon. In the hearth the metal melted into a fairly symmetrical flattened globule. The amount evaporated was 0.93 g to make foils having areal density about 473 μg/sq.cm at a distance of 20 cm from the source. The amount expected to be evaporated was 0.67 g for the source behaving as a surface source, and 1.37 g for a source behaving as a point source, to make foils of this areal density at that location. Thus the globule behaved roughly midway between a point source and a surface (Lambertian) source. For the foils of interest, we used a pieces of a slug of 99.99+% (metals' basis) Pt from Alfa Aesar [14]. This material evaporated with essentially the same emission characteristics.

High power was needed, because the Pt globule tended to make good contact with the hearth (a problem that also occurs with

evaporating aluminum from a copper hearth). The Pt did not stick tightly, and so perhaps alloying of Cu into Pt did not occur, although this has not been checked. We needed up to 175 mA emission current at 10 kV in the e-gun to get 0.05 nm/s deposition rate at 20-cm distance. At that emission current, water boiled in the cooling circuit for the electron-gun hearth.

The resulting foils were grayish, with a somewhat matte finish. We tentatively attribute this appearance to crystallinity of the KI parting agent. Adhesion to the KI underlayer was poor. These Pt foils had some bubbly regions at which the foil spontaneously released from the KI underlayer. At other places, just cutting the foil with a razor blade pulled strips of the foil off of the substrate. Nevertheless, the foils could not be floated off. The foils were so hydrophilic that they would not stay on the water surface when floating was attempted. They could not be floated by introducing droplets at scribed lines on the foil on its substrate; water would preferentially go over the exterior surface, then gradually creep underneath too. Foil pieces could be removed from the ferrotype plate by totally immersing the foil on its substrate, then peeling off the foil using tweezers, but the foils could not be made to stay on the water surface, and could not be picked up over 19-mm holes in immersed frames. Collodion-coating the foil [15] made it hydrophobic, but it would not float off; the collodion penetrated the foil and the KI layer, sticking the foil firmly to the FT plate. Attempting to rescue the foil then by rinsing the foil with methyl alcohol (which dissolves collodion) did loosen the foil, but then it could still not be floated off without immersing it totally in water, and even then the pieces could not be released satisfactorily.

We treated the foils so that they would float off, by evaporating about 60 nm of Paraflint-H1 onto the exterior surface of the foil on its substrate at room temperature. Paraflint-H1 is a high-melting-point wax often used for coating dance floors [16]. Then the foil could be droplet-floated without difficulty. When released, we transferred it to a big dish of water. Pieces of the foil tended to stay together at this point, but we could easily separate them by placing a drop of ethanol on the border between two pieces, using a glass rod [17].

We cleaned aluminum frames having 19-mm-diameter apertures by washing them with dish detergent and Comet™ [18] cleanser,

then wet-sanding them with #220 carburundum paper, rinsing them well and storing them under distilled water without permitting them to dry. Of the 14 foils successfully floated off of the ferrotype plate, every one survived pickup onto its frame. When a foil was dry, it adhered tenaciously to its frame; no adhesive was required.

We removed the Paraflint from mounted foils using a 30-minute oxygen etch [19]. There was reason to believe that this would have no deleterious effect on the Pt foils [20]. We used 30-minutes' exposure; less would have been adequate. The Paraflint coating was originally bluish-gray; etching quickly restored the plain gray color of the Pt in 5-10 minutes.

References:

[1] B.A. Movchan and Z.V. Demchisin, Phys. Met. Metallogr. (USSR) 28 (1969) 83-90.

[2] J.A. Thornton in Modeling of Optical Thin Films, M.R. Jacobson, ed., Proc. SPIE 821 (1988) 95-103.

[3] E. Graper, "Thin Film Evaporation Source Reference", in R.D. Mathis Co., 2840 Gundry Ave., Long Beach, California, 90806, U.S.A. company literature (1988).

[4] E. Hedborg, F. Winquist, and I. Lundström, Appl. Phys. Lett. 64, 420-421 (1994).

[5] J.C. Gursky and J.A. O'Rourke, Nucl. Instrum. Meth. 167, (1979) 145-149.

[6] H.J. Maier, Nucl. Instrum. Meth. Physics Research A303 (1991) 172-181.

[7] K.O. Zell, INTDS Newsletter, June 1986.

[8] Apollo Metals, Inc., 1001 14th Avenue, P.O. Box 4045, Bethlehem, Pennsylvania, U.S.A., 18018

[9] A. Méens, I. Rossini and J.C. Sens, Nucl. Instrum. Meth. Physics Research A334 (1993) 200-202.

[10] Material Evaporation Guide, Leybold-Heraeus, 6500 Fly road, East Syracuse, New York, 13057.

[11] P. Maier-Komor, "Technische Tabellen", Materials Research GmbH, Hansastr. 17, 8000 Munich 21, Germany, company literature (Feb. 1981).

[12] "Thin Film Deposition Suggestions" in Kurt J. Lesker Co., 2186 Research Drive, Livermore, California, U.S.A. company literature (1989).

[13] K.J. Ross and B. Sonntag, Rev. Sci. Instrum. 66 (1995) 4409-4433.

[14] Alfa Aesar, 30 Bond Street, Ward Hill, Massachusetts, U.S.A. 01835-8099.

[15] J.O. Stoner, Nucl. Instrum. Meth. Physics Research A362 (1995) 167-174.

[16] Moore and Munger, Inc., Two Corporate Drive, #434, Shelton, Connecticut, U.S.A. 06484.

[17] This method was suggested to the author by W. Lozowski (1990).

[18] Proctor and Gamble, Cincinnati, Ohio, U.S.A. 45202.

[19] J.O. Stoner, Nucl. Instrum. Meth. Physics Research A480 (2002) 171-177.

[20] H. Gary Pippin and R.J. Bourassa, NASA Contractor Report 4662 (June 1995), obtained from NASA, Langley Research Center, Hampton, Virginia, 23681-0001. See also: T.R. Gull, H. Herzig, J.F. Osantowski and A.R. Toft, Appl. Optics 24 (1985) 2660-2665.