Observation of double resistance anomalies and excessive resistance in mesoscopic superconducting Au_{0.7}In_{0.3} rings with phase separation

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We have measured mesoscopic superconducting Au_{0.7}In_{0.3} rings prepared by e-beam lithography and sequential deposition of Au and In at room temperature followed by a standard lift-off procedure. The majority of the samples are found to exhibit highly unusual double resistance anomalies, two resistance peaks with the peak resistances larger than the normal-state resistance, near the onset of superconductivity in the R(T) (resistance vs temperature) curves, and an h/2e resistance oscillation with a very small amplitude. A magnetic field applied perpendicular to the ring plane appears to suppress the low-temperature peak easily, but only broadens the high-temperature peak. In the intermediate-field range, the high-temperature resistance peak becomes flat down to the lowest temperature, resulting apparently in a magnetic-field-induced metallic state with its resistance higher than the normal-state resistance, referred to here as excessive resistance. The dynamical resistance vs bias current measurements carried out in samples showing double resistance anomalies suggest that there are two critical currents in these samples. We attribute the double resistance anomalies and the two critical currents to the presence of two superconducting phases originating from the phase separation of $Au_{0.7}In_{0.3}$ in which In-rich grains of AuIn precipitate in a uniform In-dilute matrix of Au_{0.9}In_{0.1}. The local superconducting transition temperature of the In-rich grains is higher than that of the In-dilute matrix. The double resistance anomalies are not found in a sample showing the conventional h/2e Little-Parks (LP) resistance oscillation, which we believe is due to the absence of the phase separation in this particular sample. Finally, we argue that the h/2e resistance oscillation observed in samples showing double resistance anomalies is not the LP but rather the Altshuler-Aronov-Spivak resistance oscillation of normal electrons enhanced by superconductivity.

DOI: 10.1103/PhysRevB.75.064509

PACS number(s): 74.78.-w, 74.81.-g, 74.40.+k

Superconductivity in the mesoscopic superconductors, which may be defined in general as superconductors with at least two of their dimensions comparable to or smaller than the characteristic lengths of the superconductor, the superconducting coherence length or penetration depth in the zero-temperature limit, has been a very active area of research in recent years. The advances in nanofabrication technologies have made it possible to fabricate devices with various geometries and material characteristics in which novel phenomena were discovered. For example, a highly unusual resistance anomaly, a resistance peak with the peak resistance larger than the normal-state resistance at the onset of the superconductivity, was found in mesoscopic zero- or onedimensional (0D or 1D), $^{1-4}$ as well as two-dimensional (2D) (Ref. 5) samples, and explained either in the picture of charge imbalance process near the superconductor-normal metal (S-N) interface^{2,3,5,6} or that of a nonuniform distribution of the current within the locus of the S-N interface.^{4,7} In both pictures, the presence of an S-N interface in the sample is necessary for the resistance anomaly to be observed.

Recent structural and electrical transport studies of Au_{0.7}In_{0.3} films revealed an interesting phase separation in which In-rich grains, most likely an intermetallic compound of AuIn, precipitate in a uniform In-dilute matrix, most likely Au_{0.9}In_{0.1}. The superconducting transition temperature of the In-rich grains is substantially higher than that of the In-dilute matrix, forming an array of superconductor-normal metal-superconductor (*SNS*) Josephson junctions⁸ and a network of *S-N* interfaces. Interesting phenomena were found in planar and cylindrical films of Au_{0.7}In_{0.3}.^{9,10} In particular, an *h*/4*e*, rather than *h*/2*e*, resistance oscillation was found in doubly

connected cylinders of Au_{0.7}In_{0.3}. The h/4e oscillation was attributed tentatively to the existence of π junctions along the circumference of the cylinder.¹⁰ In single mesoscopic rings of Au_{0.7}In_{0.3} the presence of such π junctions will lead to a phase shift of π in the h/2e oscillation if the total number of π junctions along the ring is odd.

In this paper, we report our experimental studies of mesoscopic superconducting $Au_{0.7}In_{0.3}$ rings. Conventional h/2e Little-Parks (LP) resistance oscillation was observed in one of the samples. However, whether there was a phase shift of π was not determined primarily for technical reasons (see below). Surprisingly, in the samples showing an h/2eresistance oscillation with an oscillation amplitude much smaller than that expected for LP oscillation, double resistance anomalies and a magnetic-field-induced metallic state^{11,12} with excessive resistance were found. We attribute these observations to the separation of In-rich and In-dilute phases in this type of rings, which was absent in the sample that exhibited the conventional LP effect.

Conventional *e*-beam lithography was used to prepare the Au_{0.7}In_{0.3} rings. The ring pattern was generated using double-layer PMMA/MMA resist on a polished 1×1 cm² sapphire substrate. Sequential thermal evaporation of alternating 99.9999% pure Au and In layers, with the layer thickness determined by the appropriate atomic ratio of Au to In, was carried out at ambient temperature in a conventional evaporator with a vacuum of 1×10^{-6} torr or slightly better. The ring pattern was placed with respect to the Au and In sources so as to minimize the shadow effect during evaporation. An atomic force microscope (AFM) was used to image the resulting Au_{0.7}In_{0.3} rings before and/or after the measure-

TABLE I. Parameters for Au_{0.7}In_{0.3} rings used in the present study. T_c is determined at the onset of the resistance drop for ring 6-075, and for the rest of the rings at the onset of the HTRP. *d* is diameter. Thickness $t \approx 30$ nm for all rings. The linewidth *w* is nominally 100 nm for all rings.

Sample	d (µm)	$R_{N,\Box}(\Omega)$	$\rho_N \left(\mu \Omega \text{ cm}\right)$	T_c (K)
6-075	0.75	13.5	40.6	0.450
6-100	1.00	13.5	40.4	0.380
7-100	1.00	12.5	37.4	0.370
8-075	0.75	11.3	33.8	0.359
8-100	1.00	12.4	37.2	0.355

ments. The electrical transport measurements were carried out in a dilution refrigerator, which is equipped with a superconducting magnet and has a base temperature <20 mK. All electrical leads entering the cryostat were filtered by RF filters with an attenuation of 10 dB at 10 MHz and 50 dB at 300 MHz. Resistance characteristics were measured with a dc current source and a nanovoltmeter. The magnetic field was applied perpendicular to the substrate.

Five rings of Au_{0.7}In_{0.3} were measured in this study. Their structural and electrical parameters are listed in Table I. Two rings have a diameter of 0.75 μ m and the rest have a diameter of 1 μ m. The thickness of all rings is nominally 30 nm. The linewidth of all rings is nominally 100 nm. AFM imaging found a slightly larger than expected linewidth, 130 nm for ring 6-100, which could be due to organic residues from the lift-off process. The AFM image of ring 6-100, a $1-\mu$ m-diam ring, is shown in Fig. 1(a), with a schematic shown in Fig. 1(b) to detail the various dimensions of the sample. Rings with a 0.75 μ m diameter have the same layout with a 4 μ m voltage-probe separation. Two features of the samples should be noted. First, as seen in the schematic of the sample [Fig. 1(b)], the linewidth increases slightly at the nodes of the voltage leads going towards the large contact pads. Such a variation of the linewidth, observed in all samples, was probably due to an overexposure while writing the large contact pads; second, the surface roughness as seen by AFM increases at the narrow part of the sample within the voltage leads, see Fig. 1(c)]. This roughness might be due to the contamination by the organic residues such as PMMA and/or the developer during the lift-off process, which may explain the increase of the normal-state sample resistivity, ρ_N (Table I), compared to that of the Au_{0.7}In_{0.3} films with the same thickness prepared under nominally the same conditions.⁸

In Fig. 2, normalized resistance as functions of temperature R(T) in zero magnetic field are shown for all five rings. It is clear that there exist two types of behaviors among these rings. For ring 6-075, a smooth resistive transition was seen. For the other four rings, however, two resistance peaks were found near the onset of the superconducting transition. It is seen that the low-temperature resistance peak (LTRP) is relatively sharp, with a resistance about 30–70 % higher than the normal-state resistance, R_N . The high-temperature resistance peak (HTRP) is broader and smaller in height (10–20 % higher than R_N) than LTRP.



FIG. 1. (Color online) (a) Atomic force microscope (AFM) image of a $1-\mu$ m-diam Au_{0.7}In_{0.3} ring (6-100, see Table I); (b) schematic corresponding to the AFM image in (a). The linewidth of all rings is nominally 100 nm. AFM imaging found a slightly larger than expected linewidth, 130 nm for ring 6-100, which could be due to organic residues from the lift-off process; (c) height profile along the ring arms showing surface roughness.

The parameters of our Au_{0.7}In_{0.3} rings are shown in Table I. Another important parameter, the superconducting coherence length in the zero-temperature limit, which can be used to judge whether our samples are in the mesoscopic limit, can also be estimated. Within the Ginzburg-Landau theory, the superconducting coherence length can be estimated from the upper critical field, $\xi(0) = \sqrt{3\Phi_0}/\pi w H_{c2}$, where Φ_0 =h/2e is the flux quantum and w is the linewidth, which yields a $\xi(0)$ value of 88 nm for ring 6-100 based on the experimental value of $H_{c2}(20 \text{ mK}) = 1300 \text{ Oe.}$ Alternatively, we can also estimate $\xi(0)$ using the microscopic theory for dirty superconductors, $\xi_{0,dirty} \approx 0.86 (\xi_0 l_{el})^{1/2}$, where ξ_0 $=\hbar v_F/\pi\Delta$ is the coherence length in the clean limit and l_{el} is the mean-free path.¹³ Using a gap value of Au_{0.7}In_{0.3}, Δ ≈ 0.088 meV, obtained from planar tunneling measurements on Au_{0.7}In_{0.3} films, and a mean-free path of 1.68 nm using $l_{el} = mv_F / \rho ne^2$, where m is the mass of electron, v_F is the Fermi velocity, ρ is the resistivity, and *n* is the free-electron



FIG. 2. Normalized resistances as functions of temperature R(T) for five Au_{0.7}In_{0.3} rings. Rings are labeled as indicated. Parameters for these rings are shown in Table I. The inset shows a LAMH theory fit (solid line) to R(T) data of ring 6-100 (see text).

density, we obtain a value of $\xi_{0,dirry}$ of 68 nm. Therefore it is likely that $\xi(T)$ is longer than the linewidth of the ring (nominally 100 nm) at temperatures sufficiently close to the superconducting transition temperature. Furthermore, for all samples shown in Fig. 2, the tail of the resistive transition in R(T) can well be described by the Langer-Ambegaokar and McCumber-Halperin (LAMH) theory¹⁴ of thermally activated phase slips in the 1D limit (an example of fitting is shown in the inset of Fig. 2), providing additional support that our samples were in the 1D limit.

Resistance anomaly featuring a single resistance peak at the onset of superconductivity has been widely observed in mesoscopic superconductors¹⁻⁴ as well as in 2D superconducting films.⁵ It seems reasonable that the double resistance anomalies observed in the present work and the previously observed resistance anomaly share a similar physical origin. Since the resistance anomaly was found to occur at the onset of superconductivity, the occurrence of the double resistance anomalies appears to indicate the existence of two T_c 's. In Fig. 3(b), we plot the dynamical resistance dV/dI as functions of the bias current I for ring 6-100 at different temperatures within the resistive transition as marked by arrows in Fig. 3(a). At T=0.26 K, two sharp rises in dV/dI were seen (shown by the dashed line and by the arrow, respectively, at 0.26 K), which appear to correspond to two critical currents, I_c 's. Indeed the dynamical resistance was vanishingly small below $\approx 0.05 \ \mu$ A, the smaller I_c , evolving into a central peak near zero bias current as temperature was raised to 0.30 K. At this temperature, the dynamic resistance showed a sharp rise at $\approx 0.2 \ \mu$ A, the larger I_c . While the smaller I_c was found to become zero around 0.30 K, the larger I_c survived at least up to 0.34 K, roughly where the HTRP was located. Above this temperature, the entire sample turned normal. The presence of two T_c 's and two I_c 's suggests strongly the presence of two superconducting phases in the sample.

Similar to planar $Au_{0.7}In_{0.3}$ films,⁸ these two phases should be the In-rich grains, most likely the intermetallic



FIG. 3. (a) R(T) for ring 6-100 in zero field. The temperatures at which dV/dI were taken [shown in (b)] are indicated by arrows; (b) dynamical resistance dV/dI (calculated from the dc current biased *I-V* curves using numerical derivatives) at various temperatures as indicated. Two critical currents are indicated by the dashed line and by the arrow, respectively. All curves except the one for T = 0.26 K are shifted for clarity.

compound of AuIn, and the Josephson coupled In-rich grains embedded in the uniform In-dilute matrix, most likely Au_{0.9}In_{0.1}. The superconducting transition temperatures of the In-rich grains are higher than that of the In-dilute matrix. As a result, the sample can be viewed as a random array of SNS Josephson junctions. Such an underlying structure will not only give rise to many S-N interfaces, but also lead to the nonuniform distribution of current and the creation of phase slip centers in the sample, favoring the occurrence of resistance anomaly. Despite the randomness, there exist two typical T_c 's. Therefore, HTRP should correspond to the resistance anomaly of the individual In-rich grains (with higher T_c and larger I_c) while LTRP should correspond to that of the junction array formed by the In-rich grains and the In-dilute matrix (with lower T_c and smaller I_c). Interestingly, features found in the dynamical resistance in currents below the larger $I_c ~(\approx 0.2 ~\mu A)$ and their temperature dependence, i.e., the sharp rise above the smaller I_c ($\approx 0.05 \ \mu A$) and the emergence of a central peak near zero bias current, are similar to those observed in mesoscopic Al wires showing resistance

anomaly,¹ which supports our assigning the lower T_c and smaller I_c to the overall sample.

Double resistance anomalies were observed previously.^{15,16} In one experiment,¹⁶ two resistance peaks were observed in a filled but not in an open Al square of mesoscopic size $1 \times 1 \ \mu m^2$ (our Au_{0.7}In_{0.3} rings are open) below the onset of superconductivity, not at the onset of superconductivity as seen in our Au_{0.7}In_{0.3} rings. Furthermore, the resistance peaks were only seen in a finite field and their resistance values were below the normal-state resistance. Therefore we believe that those resistance peaks were unrelated to the physics that is responsible for the double resistance anomalies observed in our Au_{0.7}In_{0.3} rings. However, the double resistance anomalies and a common feature seen in the $Au_{0.7}In_{0.3}$ rings (namely, the LTRP is always higher and sharper than the HTRP) were found in one of the granular films prepared from preformed In clusters embedded in a Kr matrix¹⁵ at large excitation currents. Obviously, many differences exist between the two very different systems, which may be responsible for the presence of triple resistance anomalies found in In-Kr films in small excitation currents that were not observed in our Au_{0.7}In_{0.3} rings. It should be noted that the current dependence of these resistance anomalies, in which the higher temperature peak was suppressed first by increasing excitation current, is very different from that in the Au_{0.7}In_{0.3} rings. Similar to our $Au_{0.7}In_{0.3}$ samples, this film can be modeled as an array of superconductor-insulator-superconductor Josephson junctions,¹⁵ with a T_c smaller than that of the individual In clusters.

The measurements on the magnetic-field dependence of the double resistance anomalies, shown in Fig. 4, provide further support to the picture that the presence of two superconducting phases is responsible for the double resistance anomalies. It is seen that LTRP was affected significantly at a field as low as 100 Oe, becoming barely visible at 300 Oe, which appears to be a critical field, H_c . On the other hand, in a field as high as 900 Oe, even though HTRP was broadened and shifted to lower temperatures, it was clearly visible. In fact, even at a field up to 1300 Oe, HTRP could still be identified close to the lowest temperature, 20 mK. It is therefore evident that the HTRP was associated with In-rich grains with a critical field of 1300 Oe while LTRP belongs to the *SNS* Josephson junction array with a critical field of 300 Oe.

The dynamical resistance curves taken at 300 Oe [Fig. 5(a)] show that, at 0.1 K, the minimum near the zero bias current was replaced by a peak, indicating that the critical current of the SNS Josephson junction array was essentially zero at 300 Oe. This is consistent with the results of Fig. 4, which in turn supports the assessment that LTRP was associated with the *SNS* Josephson junction array. The sharp rises in dV/dI at high bias current, on the other hand, were clearly visible at H=900 Oe [Fig. 5(b)], suggesting that HTRP was associated with the In-rich grains.

An interesting feature emerging from Fig. 4 is that, in the intermediate field range, the broadened HTRP extended to the lowest temperature we measured. At H=900 Oe, in particular, the resistance became independent of temperature down to 20 mK, with a resistance value larger than that of



FIG. 4. R(T) at several magnetic fields as indicated for ring 6-100. The fields were applied perpendicular to the plane of the ring. A resistance plateau higher than R_N is seen down to the lowest temperature available (<20 mK) for H=300 and 900 Oe.

the normal-state resistance, R_N . The existence of this lowtemperature resistance plateau, referred to here as excessive resistance, appears to suggest the existence of a metallic state in which the In-rich grains were superconducting, but not Josephson coupled.^{11,12} This metallic state, with its onset temperature >0.2 K, might not be due to heating as it is generally believed that only below 0.1 K electron dephasing and heating from the coupling to the environment start to be a serious concern. The observation of a metallic state with its resistance larger than R_N and its onset temperature as high as 0.2 K may be taken as an evidence that this metallic state is intrinsic. The physics of such a metallic state is yet to be explored.

For ring 6-075 with a smooth R(T) showing no double resistance anomalies, its onset T_c was around 0.45 K, close to that observed in thinnest planar films (thickness ≤ 15 nm) of Au_{0.7}In_{0.3}.⁸ In those thinnest films, the interdiffusion of Au and In was suppressed by the close proximity to the substrate, resulting in films that were uniform rather than phase separated. Ring 6-075 might be of similarly uniform structure. This assessment is consistent with the observation of conventional h/2e LP resistance oscillation in this sample as shown in Fig. 6(a). Unfortunately, whether there was a phase shift of π in the h/2e oscillation in ring 6-075, the question that motivated this work originally, could not be determined because it is difficult to determine the flux sufficiently precisely because of the possibility of trapping flux in the superconducting magnet.

For the four rings showing double resistance anomalies, a



FIG. 5. (a) Dynamical resistance dV/dI at various temperatures, as indicated, for ring 6-100 at H=300 Oe; (b) dV/dI at H=900 Oe. dV/dI curves are based on numerical derivatives of dc current biased *I-V* measurements. All curves except the one at T=0.10 K are shifted for clarity in both panels.

weak h/2e resistance oscillation was found as shown in Figs. 6(b) and 7 for ring 6-100. The oscillation was most pronounced in the intermediate-field range, and weaker or even absent in the low-field range. For example, for the R(H) curve at T=0.3 K, the oscillation was pronounced between 350 and 600 Oe, was less obvious between 100 and 300 Oe, and indiscernible below 100 Oe (H < 0 data not shown for clarity). A Fourier analysis of the data with different field ranges generated similar findings. The oscillation was completely quenched above H_c or T_c .

It is of interest to ask whether the h/2e resistance oscillation observed in these rings showing double resistance anomalies is LP resistance oscillation. Anomalous LP oscillation was observed in Al loops in a previous study in lowfield range, where the oscillation was distorted due to the complication of resistance anomaly and restored to the conventional LP with an increasing field.¹⁷ However, we believe that the oscillation in the current work is unlikely to be related to the LP effect. First, the resistance oscillation was absent in low fields, inconsistent with the LP behavior; second, the amplitude of the resistance oscillation is very small in comparison with the conventional LP resistance oscillation



FIG. 6. (Color online) (a) LP resistance oscillation of ring 6-075 at various temperatures as indicated. The period of the oscillation corresponds to h/2e; (b) AAS resistance oscillations enhanced by superconductivity (see text) of ring 6-100 at various temperatures as indicated. The oscillation period corresponds to h/2e. The resistance variation corresponding to $20e^2/h$ is shown.

shown in Fig. 6(a). The oscillation amplitude is less than 5 Ω for ring 6-100 but as large as 50 Ω in ring 6-075. For both samples, their zero-temperature superconducting coherence lengths estimated from the upper critical-field value or the formula for dirty superconductors as shown above are very close. The difference in diameter alone cannot explain the large difference in amplitude since the LP amplitude is proportional to $[\xi(0)/d]^2(\Delta R/\Delta T)$, where d is the ring diameter and $\Delta R / \Delta T$ is the measured slope in the transition region;¹³ Our Au_{0.7}In_{0.3} rings consist of a random SNS junction array because of the phase separation, which will give rise to three temperature regimes at given applied flux values. At the lowest temperatures the whole ring will be superconducting while at sufficiently high temperatures the ring will be normal. In the intermediate temperatures, however, only the Inrich grains will be superconducting, with no global phase



FIG. 7. Resistance as a function of applied magnetic field R(H) at several temperatures, as indicated. The period of the resistance oscillations is h/2e. The negative magnetoresistance in the intermediate fields is related to the suppression of the resistance anomalies by the field. All curves except the one at T=20 mK are shifted for clarity.

coherence. LP resistance oscillation results from the modulation of the phase boundary of the fully superconducting phase (with global phase coherence) by the applied flux. For our Au_{0.7}In_{0.3} rings featuring double resistance anomalies, the resistive transition as a function of magnetic flux at this phase boundary is extremely sharp as shown in Fig. 7, making it difficult to observe the LP resistance oscillation. In this regard, it will be interesting to prepare rings of a lithographically defined, regular *SNS* junction array with a less sharp resistive transition to compare with our Au_{0.7}In_{0.3} rings. For our Au_{0.7}In_{0.3} samples, superconductivity in individual Inrich grains is suppressed over a rather wide range of magnetic field, in which the h/2e resistance oscillation was observed away from the boundary of the fully superconducting phase.

We believe that the h/2e resistance oscillation in rings showing double resistance anomalies is the Altshuler-Aronov-Spivak (AAS) resistance oscillation¹⁸ enhanced by superconductivity. The AAS effect is a result of the coherent backscattering of normal electrons in disordered systems, as first experimentally seen by Sharvin and Sharvin.¹⁹ AAS oscillation was shown to be enhanced in normal metal samples in contact with one or more superconducting islands in several experiments.^{20,21} The amplitude of AAS resistance oscillation is determined by both L (the length of the circumference of the time-reversal path) and L_{φ} (the electron dephasing length), in the form $\propto \exp(-L/L_{\omega})$. In our experiment, when In-rich grains became superconducting, L, which included only the normal part of the ring, decreased. Meanwhile, L_{φ} tends to increase when superconducting regions are present.²¹ As a result, the amplitude of the resistance (or conductance) oscillation was enhanced as $\Delta G \approx 11e^2/h$ for ring 6-100. (Here an effective normal-state resistance of the ring itself, 105.8 Ω , estimated from the sample geometry, rather than the total resistance of the sample, 510 Ω , is used.) Such an enhanced amplitude is comparable to previous observations.^{20,21} The AAS effect enhanced by superconductivity also explains the fact that the oscillation was quenched above T_c or H_c [Figs. 6(b) and 7]. The AAS oscillation, without being enhanced by superconductivity, has an amplitude on the order of $\Delta G \approx e^2/h$, corresponding to a resistance variation of 0.5 Ω for ring 6-100, too weak to be observed in our measurements.

In closing, we would like to discuss briefly the possible physical origin of the double resistance anomalies. As mentioned above, the double resistance anomalies should be due to a physical process similar to the resistance anomaly observed previously in mesoscopic superconducting samples. The physical origin of the resistance anomaly is still subject to an intensive debate¹⁻⁷ and is not settled down. One school of thought has argued that the charge imbalance near the S-N interface contributes to the resistance anomaly.^{2,3,5,6} The S-N interface can be a result of either phase slip centers at weak links or sample inhomogeneity. Superconducting voltage probes are required to observe the anomaly.⁵ The other school has pursued an alternative model based on the nonuniform distribution of the current across the sample within the locus of the S-N interface.^{4,7} The S-N interface originates from sample inhomogeneity and nonsuperconducting voltage probes can also lead to the resistance anomaly. Even though both scenarios described above can qualitatively explain some of our experimental observations, a quantitative comparison between the theory and experiment appears not possible with the information available to us.

We would like to acknowledge useful discussions with B. Pannetier. This work was supported by NSF under Grant No. DMR-0202534.

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