Vortex–glass transformation within the surface superconducting state of $\beta$-phase Mo$_{1-x}$Re$_x$ alloys

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Abstract
We have performed an experimental study on the temperature dependence of electrical resistivity $\rho(T)$ and heat capacity $C(T)$ of the Mo$_{1-x}$Re$_x$ ($x = 0.20, 0.25$) alloy superconductors in different magnetic fields. In the presence of applied magnetic field, the electrical resistivity of these alloys goes to zero at a temperature well above the bulk superconducting transition temperature obtained with the help of heat capacity measurements in the same magnetic field. Our study indicates the presence of a surface superconducting state in these alloys, where the flux lines are pinned in the surface sheath of the superconductor. The configuration of the flux lines (two-dimensional pancake-like) in the surface sheath is understood in the realm of the flux-spot model. Experimental evidence in support of the surface mixed-state state or ‘Kulik vortex-state’ and the occurrence of a vortex–liquid to vortex–glass transition is presented.

Keywords: vortex–glass state, surface mixed state, surface superconductivity, electrical resistivity, metals and alloys

(Some figures may appear in colour only in the online journal)

1. Introduction

The phenomenon of surface superconductivity was discovered by Saint-James and de Gennes back in 1963 [1, 2]. They showed that for any finite size sample, the nucleation of the superconducting region easily occurs near the sample surface in the case of a metal–insulator (or metal–vacuum) interface, in the presence of a parallel magnetic field $H_{c3}$ higher than the upper critical field $H_{c2}$ by a factor of 1.695. They also argued that the nucleation field $H_{c3}$ for surface superconductivity may be strongly modified by the normal metal coating (e.g. Cu, Ag) at the sample surface [3, 4]. With the discovery of surface superconductivity, it was possible to explain a large amount of experimental data, which had previously been discarded as due to sample inhomogeneity [5]. Later, many researchers studied the surface superconducting state in many superconductors such as Pb-Tl, Nb, MgB$_2$ [6–12]. Initially it was thought that for surface superconductivity (and surface critical current) to exist, the local magnetic field needs to be parallel to the sample surface [7, 13–16]. Subsequent work, however, showed that surface superconductivity (and surface critical current) may be observed even when the local magnetic field has a nonzero perpendicular component arising because of the misalignment of the applied magnetic field relative to the sample surface, due to the magnetic field related to an applied transport current, the demagnetization factor and the local roughness of the sample surface [17]. It was inferred [17] that the
perpendicular component of the magnetic field \( (H > H_{c2}) \) may penetrate the sample surface as an array of quantized flux spots \( \Phi_0 = \frac{h}{2e} \approx 2.0678 \times 10^{-15} \) Wb whose free energy is sensitive to the local properties of the surface sheath of the superconductor. Since the local properties of the surface sheath will vary spatially, the free energy of the flux spots will also vary spatially and the flux spots will stay pinned at the locations of minimum free energy (see figure 1) [17].

The pinning of the flux spots depends on the degree of surface roughness, and becomes increasingly significant till the scale of roughness becomes comparable with the spacing between the flux spots approximately [17]. This suggests the existence of a dense quantized two-dimensional (2D) flux lattice (or flux spots) in the surface region of a superconductor [17]. The absence of surface pinning or the presence of a driving force (due to a transport current in the presence of magnetic field) greater than the pinning strength, allows the array of flux spots to move freely in the surface superconducting region producing a non-zero voltage and a vanishing surface critical current [17].

Polycrystalline samples of Mo\(_{1-x}\)Re\(_x\) \((x = 0.20, 0.25)\) alloys were prepared by melting molybdenum and rhenium (99.95% purity) in an arc furnace under high purity (99.999%) argon atmosphere. The samples were flipped and re-melted six times to ensure homogeneity. X-ray diffraction (XRD) measurement shows that the samples form in the single phase bcc crystal structure (the XRD results are presented elsewhere) [36]. The electrical resistivity of the Mo\(_{1-x}\)Re\(_x\) alloy samples was measured in the temperature range 1.5–15 K and up to 2 T magnetic field using the standard four probe technique with the help of a superconducting magnet cryostat (Oxford Instruments, UK) system. The temperature dependence of electrical resistivity in 0, 0.5, and 1 T magnetic fields were also measured after applying a Cu coating on the Mo\(_{0.75}\)Re\(_{0.25}\) alloy and a Ag coating on the Mo\(_{0.80}\)Re\(_{0.20}\) alloy. The Cu coating on the Mo\(_{0.75}\)Re\(_{0.25}\) alloy was applied using the electroplating technique (with the help of a CuSO\(_4\) solution). Some portions of the Cu coating were then removed by filing for placing the four probe electrical contacts. The Ag coating on the Mo\(_{0.80}\)Re\(_{0.20}\) alloy was applied by dipping the sample in electrically conducting Ag paint after placing the electrical contacts for the four probe technique. A constant current of 100 mA was used to measure the resistivity of these alloys. Heat capacity measurements were performed in the temperature range 2–15 K in various magnetic fields up to 3 T using a Physical Property Measurement System (PPMS; Quantum design, USA).
3. Results and discussion

Figure 2 shows the temperature dependence of electrical resistivity in zero and different applied magnetic fields. The zero field superconducting transition temperatures \( T_c \) for the present alloys have been reported elsewhere [36] and they are in good agreement with previously reported values [37]. The width of the superconducting transition \( \Delta T_c \) for different field values was estimated as the temperature difference between 10% and 90% of the normal state resistivity \( \rho_N \).

In figure 3, the \( \Delta T_c \) as a function of magnetic field is plotted, which is well described using a linear relation:

\[
\Delta T_c (H) = \Delta T_c (0) + kH
\]

where \( \Delta T_c (0) \) is the width of the superconducting transition in zero field and \( k \) is a constant. The presence of multiple superconducting phases in a sample (and the resulting spatial distribution of \( T_c \)) has often been considered to be a possible reason behind the broadening of the normal-to-superconducting phase transition [38, 39]. The fitting of equation (1) to the \( \Delta T_c \) versus \( H \) data, however, negates this possibility. A sample with multiple superconducting phases would have shown an upturn (or curvature) in the low field side of the \( \Delta T_c \) versus \( H \) plot due to the different field dependencies of \( T_c \) in the different superconducting phases [38]. Moreover, XRD and optical metallography studies of both the Mo\(_{1-x}\)Re\(_x\) \((x = 0.20, 0.25)\) alloys [36] do not indicate the presence of a second phase.

Apart from the existence of multiple superconducting phases in the sample, the broadening of the superconducting transition may also be due to the presence of multiple superconducting gaps [40, 41], vortex–glass state [42, 43], vortex-melting behaviour [41] and surface superconductivity [44, 45]. In this context, surface superconductivity has already been reported in Mo\(_{1-x}\)Re\(_x\) alloys [34]. On the other hand, the superconducting properties of \( \beta \)-phase Mo\(_{1-x}\)Re\(_x\) alloys are significantly influenced by the presence of two superconducting energy gaps [31]. To investigate the properties of surface superconductivity, \( \rho (T) \) and \( C(T) \) curves were plotted in the same temperature window in zero field (figures 4(a) and (c)) as well as in a higher magnetic field \((> H_{c1})\) (figures 4(b) and (d)). In all the panels of figure 4, the (blue) solid circles (dots) represent the \( C(T) \) measured in different \( H \) (specified in each of the panels), the (black) solid triangles represent the \( \rho (T) \) measured before coating the samples with Cu or Ag, and the (red) open squares represent the \( \rho (T) \) measured after applying these metal coatings. Figures 4(a) and (c) show that in zero field, the signature of the superconducting transition in the \( \rho (T) \) and \( C(T) \) curves is observed at the same temperature approximately, and this signature in the \( \rho (T) \) curves is not affected by the presence or absence of the metal coatings on the samples. In the presence of applied magnetic field greater than \( H_{c1} \) (figures 4(b) and (d)), the signature of this phase transition in the not-coated
samples is observed at a higher temperature in the $\rho(T)$ as compared to that in the $C(T)$. However, after applying the metal coatings, in the presence of the same $H$, the signature of this phase transition in the $\rho(T)$ is observed to move towards lower temperatures and closer to that observed in the $C(T)$. The (four) probes used for the $\rho(T)$ measurement are on the sample surface, hence, this measurement is influenced by surface related phenomena. The suppression of the normal to superconducting transition temperature observed in $\rho(T)$ by applying a metal coating strongly suggests that this transition in the presence of magnetic field is indeed due to surface superconductivity [3, 4]. The $C(T)$ measurement, on the other hand, is influenced mainly by bulk phenomena. Figure 4 thus indicates that the superconducting transitions corresponding to the bulk and surface are distinctly different in the present alloys.

In this investigation, the focus is on the superconducting transition and its broadening observed with the help of the $\rho(T)$ measurements in the presence of applied magnetic fields. The present results (figures 2, 4), in conjugation with the flux-spot model (figure 1) [17], suggest that the nature of the mixed state formed within the surface sheath might give rise to broadening of the superconducting transition in these alloys. This is supported by the analysis of $\rho(T)$ curves in different fields, presented below.

In zero magnetic field the superconducting transition is quite sharp ($\Delta T_c < 0.1$ K) for both the Mo$_{1-x}$Re$_x$ alloys. However, the superconducting transition is increasingly broadened with the increase of applied magnetic field. Additionally, the superconducting transition in the presence of magnetic field exhibits a rounding-off behaviour near the onset of the superconducting transition and a tailing effect near completion. The rounding-off behaviour of $\rho(T)$ is possibly related to superconducting fluctuations [46]. The tail region of the superconducting transition becomes more significant as we go to higher magnetic fields, with a gradual suppression of the temperature corresponding to zero resistivity. Similar features have been observed in many bulk systems such as: iron based superconductors, SmFeAsO$_{0.85}$ [47], BaFe$_2$As$_2$ [48]; high-$T_c$ superconductors [49, 50]; and even in some of the low $T_c$ superconductors, Nb thin films and Nb/Cu superlattices [51, 52], narrow strips of Nb [53]. In film [54], amorphous Mo$_2$Si$_{1-x}$, and Mo$_2$Si films [55, 56]. Both the tailing-off behaviour as well as the broadening of the superconducting transition are generally attributed to the effect of thermal fluctuations in the superconductors, and it gives rise to a rich variety of flux-line dynamics in the presence of quenched disorder and other defects [25]. Generally, the effect of thermal fluctuations is expected to be small in the case of the low-$T_c$ superconductors. However, the effect of
shows a power law behaviour in $r = H_T T^2_H U$ in different magnetic fields for the Mo$_{1-x}$Re$_x$ alloys. The Ginzburg number for the Mo$_{1-x}$Re$_x$ alloys was estimated using the following relation [57]:

$$G_t = \frac{32\pi^2 \left( k_B T_c \kappa \lambda_{GL}(0) \right)^2}{\Phi_0^2}$$

(2)

where $k_B$ is the Boltzmann constant, $T_c$ is the superconducting transition temperature, $\kappa$ is the Ginzburg–Landau parameter, $\lambda_{GL}(0)$ is the Ginzburg–Landau penetration depth at absolute zero temperature and $\Phi_0$ is the flux quantum. To estimate the value of the Ginzburg number using equation (2), we have used $\kappa = \frac{H_2(0)}{2\pi R(0)}$. The values of $H_2(0)$ (the upper critical field at absolute zero temperature) have been taken from [36]. The thermodynamic critical field, $H_c(0)$, and $\lambda_{GL}(0)$ are estimated from the heat capacity measurements and using $\lambda_{GL}(0) = \kappa \times \xi_{GL}(0)$, where $\xi_{GL}(0) = \left( \frac{\Phi_0}{8\pi R(0)} \right)^{1/2}$, respectively. In this context, it may be noted that while the Ginzburg number generally comes out to be $10^{-2}$–$10^{-4}$ for high-$T_c$ superconductors [57–59], for low-$T_c$ superconductors this number is found to be in the range of $10^{-6}$–$10^{-8}$ [59]. Thus, the Ginzburg number for the Mo$_{1-x}$Re$_x$ alloys is similar to those of conventional low-$T_c$ superconductors, indicating that the effect of thermal fluctuations may not be substantial in these alloys. In view of the experimentally observed signature of surface superconductivity in the present Mo$_{1-x}$Re$_x$ alloys, we believe that the formation of 2D pancake-like flux lines (Kulik vortex-state [22, 23]) within the surface sheath causes an enhancement of the effect of the thermal fluctuations in these alloys. Therefore, similar to other systems [60, 61], analysis of the $\rho(T)$ behaviour is performed using the thermally activated flux-flow (TAFF) model [62].

In the TAFF model, the temperature dependence of electrical resistivity is described by the Arrhenius relation [62]

$$\rho(T) = \rho_0 \exp \left[ -U(H, T)/T \right]$$

(3)

where $\rho_0$ is the pre-exponential factor independent of field and $U(H, T)$ is the activation energy. The Arrhenius relation suggests that $\ln \rho$ versus $1/T$ should be linear in the TAFF region.

Figure 5 shows the Arrhenius plots for $\rho(T)$ in different magnetic fields for the Mo$_{1-x}$Re$_x$ ($x = 0.20$, 0.25) alloys. The activation energy ($U_0$) is estimated by fitting a straight line (blue) to the experimental data in the TAFF model. The fitted straight line (blue) in both panels of figure 5 shows a deviation from linearity at temperature $T^*$. In figure 5, the insets to panels (a) and (b) show the activation energy $U_0(H)$. For both the alloys, the $U_0(H)$ shows a power law behaviour ($U = AH^{-\alpha}$) with $\alpha \geq 2$. High values of $\alpha$ have also been reported in other superconductors, such as iron pnictide and cuprates, and has been described in terms of the collective flux-line pinning behaviour [49, 63, 64].

To analyze the experimental data below $T^*$, we have estimated the activation energy, $U(T)$, using equation (3):

$$U = -d(\ln \rho)/d(1/T)$$

(4)

The temperature dependence of the activation energy of the Mo$_{1-x}$Re$_x$ alloys is shown in figure 6. The activation energy increases rapidly with decreasing temperature below a characteristic temperature $T^*$, which matches exactly with that pointed out in figure 5 as the temperature corresponding to the deviation from linearity. The rapid increase of activation energy below $T^*$ marks the entry into a critical regime associated with the vortex–liquid to vortex–glass transformation, as previously observed in other superconductors [47, 49, 65]. Figure 7 shows the temperature dependence of inverse logarithmic derivative of resistivity with the values of $T^*$ marked by arrows. Thus, to investigate the existence of the vortex–glass state, we need to study the temperature dependence of resistivity below the characteristic temperature $T^*$.

According to vortex–glass theory [25], the electrical resistivity vanishes at the glass transition temperature $T_g$,

$$\rho \propto (T - T_g)^\delta$$

(5)

where $\delta$ is the critical exponent, defined as, $\delta = \nu(z + 2 - d)$, $\nu$ is the static exponent, $z$ is the dynamic exponent and $d$ is a
The glass temperature $T_g$ is estimated by applying the following equation to the tail region of the $r$ curves:

$$\left(\frac{d\ln \rho}{dT}\right)^{-1} = \frac{1}{s}(T - T_g)$$

(6)

According to equation (6), the inverse logarithmic derivative of resistivity is linearly proportional to the temperature of measurement. Figure 7 shows that the resistivity in the temperature range $T_g < T < T^*$ is well described by equation (6). In figure 7, the slope of the straight line fitted to the experimental data (within the temperature range $T_g < T < T^*$) gives the value of the critical exponent and its intercept on the temperature axis gives the value of the glass transition temperature $T_g$. In figure 7, the deviation from linearity at temperature $T^*$ marks the upper limit of the critical region associated with the vortex–glass to vortex–liquid phase transformation. Figures 6 and 7 show that the $\rho(T)$ in the temperature range, $T_g < T < T^*$, is well described by the vortex–glass model.

The magnetic field dependence of the critical exponents is shown in figure 8. The critical exponents increase with increasing magnetic field for both the alloys. As seen in figure 8, the maximum attainable values of the critical exponents for the $x = 0.20$ and $x = 0.25$ alloys are about 1.4 at $H = 1.2$ T and 2.4 at $H = 1.8$ T respectively. According to the vortex–glass model [59], the critical exponent values are in the range 2.7–8.5 for the 3D vortex–glass state [47]. On the other hand, the maximum permissible value of the critical exponent for the 2D vortex–glass state is reported to be 2.7 [47]. The small values of critical exponent in the present alloys indicate that the vortex–glass state has formed in the surface sheath (Kulik vortex-state).

It may be noted that in the literature the small value of the critical exponent has also been attributed to the Bose-glass phase. The Bose-glass phase is formed due to the interaction of the flux lines with correlated disorder, such as the twin boundaries and the columnar defects [59, 66, 67]. However, optical metallography studies and the published literature on these alloys [36] do not show any indication of the presence of correlated disorder (twin boundaries, columnar defects).
4. Summary and conclusion

In summary, the temperature dependence of electrical resistivity and heat capacity were measured experimentally, and the signature of vortex–liquid to vortex–glass transition within the surface mixed state or Kulik vortex-state in the Mo$_{1-x}$Re$_x$ superconductors. The onset of the bulk superconducting transition was estimated from the $C(T)$ measurements in different magnetic fields. The $T_{C\text{onset}}(H)$ values for the bulk superconducting transition are defined as the temperature where the superconducting transition just starts (onset of the peak in $C(T)$). We observe in figure 9 that all aspects related to the vortex–liquid to vortex–glass transformation take place above the bulk superconducting transition temperature. This phase diagram thus provides additional evidence of the formation of the Kulik vortex-state.

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