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FORMATION, PROPERTIES, AND ION IRRADIATION EFFECTS OF HEXAGONAL STRUCTURE MoN THIN FILMS

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Abstract

Thin films (100–120 nm) of hexagonal structure MoN have been fabricated by reaction of Mo films in an NH_3 atmosphere. The as-formed films possessed superconducting transition temperatures $T_c \approx 13$ K, with resistance ratios $r=R(296K)/R(T_c)$ in the range 5 to 10, low-temperature normal state resistivities $\rho_0 \approx 4$ to 10 μΩ-cm, and extrapolated upper critical fields $H_{c2}(0) \approx 4.0$ to 5.0 T. Thin film X-ray diffraction patterns revealed no visible second phase, with measured lattice parameters close to literature values. The effects of lattice disorder on the superconducting and electronic properties were investigated by irradiation with nitrogen ions of energy 45 and 340 keV, resulting in a nearly uniform damage profile without the introduction of any new chemical species. The results indicate that ordered hexagonal MoN shows some of the unusual properties characteristic of moderate-to-high T_c transition metal compounds, but is relatively insensitive to degradation of the superconducting properties by lattice disorder. For ion fluences Φ up to 2×10^{16} N-ions/cm^2, T_c is found to decrease monotonically and saturate at 9.5 K, almost 3/4 the initial value, while $H_{c2}(0)$ undergoes a gradual increase to 11T.

Introduction

Many compounds with high superconducting transition temperatures T_c are known to be sensitive to the degree of atomic lattice disorder, which may be imperfect stoichiometry, or defects resulting from growth conditions or from damage due to energetic particle irradiations. These latter effects have been studied extensively in the high T_c materials having the A15 structure[1] but less systematically in other classes of compounds that also possess attractive superconducting properties.[2]

Recently, there has been renewed interest in the transition metal carbides, nitrides, and carbonitrides, not only from the standpoint of practical conductor and device development,[3,4] but also as model systems for assessment of fundamental microscopic mechanisms in superconductivity.[3,5,6] Certain of these materials with the B1 (rocksalt) structure (NbN, Nb(CN), and (NbTi)N) have transition temperatures approaching 18 K, exceeded only by a few A15-structure compounds.

Recently, a theoretical prediction that B1-structure MoN should have a high $T_c \approx 30$ K has aroused interest.[7] To date, however, both experimental and theoretical efforts have indicated the stable, stoichiometric structure to be a hexagonal phase related to the WC structure.[8,9]

Because hex MoN is a moderate T_c material, stable at the stoichiometric 1:1 composition, we have initiated efforts to fabricate high quality thin films and investigate the effects of lattice disorder produced by N-ion irradiations. These preliminary results are presented in the following, and are assessed in terms of models previously applied to A15 materials, as well as some recent theoretical developments involving the effects of weak electron localization on the superconducting properties of disordered materials.[10,11]

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Experiment

A series of samples were prepared in thin film form by developing and utilizing a three step process: First, the formation of molybdenum metal thin films; second, the purification of these films; and third, the reaction with ammonia to produce hexagonal MoN.

In the first step, a molybdenum metal source was electron-beam evaporated onto single crystal sapphire substrates that were masked to yield narrow Mo strips of typical dimensions 2.5 cm long by 0.159 cm wide by 120 nm thick. During evaporation the substrate temperature was about 350 C and the system pressure in the range 1×10^{-4} Pa, which resulted in rather low-purity films, as evidenced by measured resistance ratios $r \approx 1.5$ to 3.5.

The second step consisted of a high-temperature degassing treatment at temperatures in the range 1550 to 1650 C, at pressures 2 to 7×10^{-7} Pa, and for times 10 to 20 min. For this procedure the Mo thin film samples were heated radiantly by placement inside a cylindrical susceptor, made of either Ta or Mo, heated by radio-frequency induction. The overall effect of the degassing was to markedly purify the films, resulting in measured $r \approx 30$ to 90.

In the final fabrication step the UHV chamber was backfilled with about 1000 Torr of pure NH_3, and the purified Mo films heated to 750 C, for time periods in the range three to six hours. This diffusion-limited reaction technique has been utilized previously, and found to produce layered, two-phase material—with a hexagonal MoN layer near the surface, and B1-structure Mo_2N lying below.[12] We utilized a rotating-sample thin film X-ray diffractometer to provide lattice structure characterization, and have found that, for practical reaction times of a few hours, Mo film thicknesses $d_{Mo} < 110$ nm result in exclusively single-phase, polycrystalline hexagonal MoN films.

The superconducting transition temperature T_c, temperature-dependent resistivity $\rho(T)$, and upper critical magnetic field $H_{c2}(T)$ (up to 8T, $\perp$ to the film plane) were measured using standard four-terminal techniques.

Ion irradiations of the samples were carried out at ambient temperature using total fluences Φ comprised of 45 and 340 keV N-ions in a 1:15 ratio. In this combination the damage energy deposition profile was uniform within a standard deviation $\sim 5\%$ over a 120 nm depth, and given by the mean value, $\Delta E_a/\Delta\Phi = 9.8 \times 10^{-15}$ eV/atom per N-ion/cm^2.[13] Thus one may anticipate that fluences of the order 10^{15} N-ions/cm^2 are required to produce one displacement/atom, and in our preliminary study we have used Φ in the range 1×10^{14} to 2×10^{16} N-ions/cm^2.

Results and Discussion

In Table 1 we briefly summarize the observed properties of several as-formed MoN films. Here, the superconducting transition temperature T_c is defined by the midpoint, and ΔT_c by the 10-to-90% breadth of the resistive transition, while ρ_0 is the electrical resistivity just above T_c. The resistivity ratios r are seen to fall in the range 5 to 10, indicating the high quality of these samples when compared to other transition metal nitride films prepared by similar[5] or different[6] techniques.

Table 1. Properties of as-formed hex MoN films

MoN Sample	T_c(K)	ΔT_c(K)	r	ρ_0(μΩ-cm)
21	13.17	0.26	10.2	4.9
31	12.87	0.30	5.2	11.6
41	13.07	0.22	9.7	5.0
51	12.92	0.50	4.7	12.7
186-6	13.06	0.32	5.7	9.1
FSU-1	13.07	0.46	6.4	8.7

Measured lattice constants for the present films were grouped around the values a=0.572 nm and c=0.561 nm, to within the experimental error of about ±0.0006 nm. Cell dimensions of the B1 nitrides are known to depend sensitively on composition.[8] As far as we know this dependence has not been investigated systematically for hex MoN, so we cannot be certain that our samples are at the stoichiometric composition, aside from an argument based on the low ρ_0.

The effects of ion irradiation damage on the superconducting transition temperature are shown in Fig. 1, where we have plotted T_c, reduced with respect to T_{c0} of the unirradiated samples. For comparison, we include our recent results for ambient temperature N-ion irradiations of B1-structure VN,[14] with T_{c0}=8.7 K, as well as data from the literature for irradiations of Nb_3Sn with 25 MeV oxygen-ions at $T < 30$ K,[15] and with 2 MeV He-ions at ambient T.[16] (For Nb_3Sn, the representative points shown here are taken from smooth curves drawn through the corresponding experimental data.) This latter material, for which T_{c0} = 18 K, shows radiation damage effects that are typical of high-T_c A15-structure superconductors. In order to compare the data for the different ion species and irradiated materials in a meaningful way, we have converted fluence to the deposited damage energy/atom, E_a.[13]

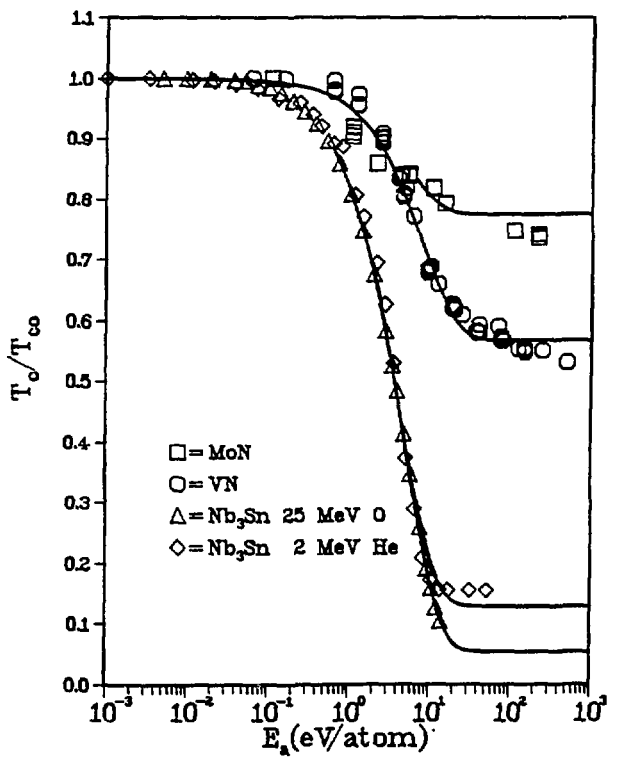

Figure 1. The transition temperature T_c, normalized by T_{c0} of the unirradiated material, as a function of the damage energy E_a deposited in the lattice by the irradiating ions.

Perhaps the most striking feature of Fig. 1 is the relative insensitivity of T_c to degradation by disorder in hex MoN and B1 VN when compared to the A15 material. It can be seen that the total reductions in T_c for MoN, VN, and Nb_3Sn are about 27%, 42%, and 84%, respectively.

Some questions remain regarding the detailed mechanisms through which T_c is suppressed by disorder in the extensively-studied A15's; but one theoretical description[17] has been fairly successful for most of these high-T_c materials that have a large, sharply-peaked N(E) at E_F (Nb_3Ge is an exception). In this treatment T_c is directly correlated with ρ_0 through a reduction in $N(E_F)$ (and consequently in the electron-phonon interaction parameter λ_{ep}), that arises from electron lifetime averaging-over and broadening of the sharp peak. This is an interesting notion when applied to the three materials of Fig. 1, where for Nb_3Sn E_F is situated at such a peak in N(E),[18] for VN E_F sits on the side of a peak,[19] and for hex MoN E_F lies close to a local minimum.[20] An analysis of this type for VN will be discussed elsewhere,[21] but here we show that this T_c-reduction mechanism is not viable in the case of hex MoN. However, recent theoretical developments in the effects upon T_c of weak electron localization appear to be applicable[10,11] and are treated briefly as well.

We find that the residual resistivity $\rho_0(\Phi)$ rises rapidly and approaches saturation near a value $\rho_0(\infty) \approx 130$ μΩ-cm. This behavior parallels that of $T_c(\Phi)$, as shown in Fig. 2, where we plot T_c as a function of ρ_0 and find a nearly linear relation except for some additional reduction of T_c near the saturation values. We find T_c = 13.2 K for pure hex MoN by extrapolating to ρ_0 = 0.

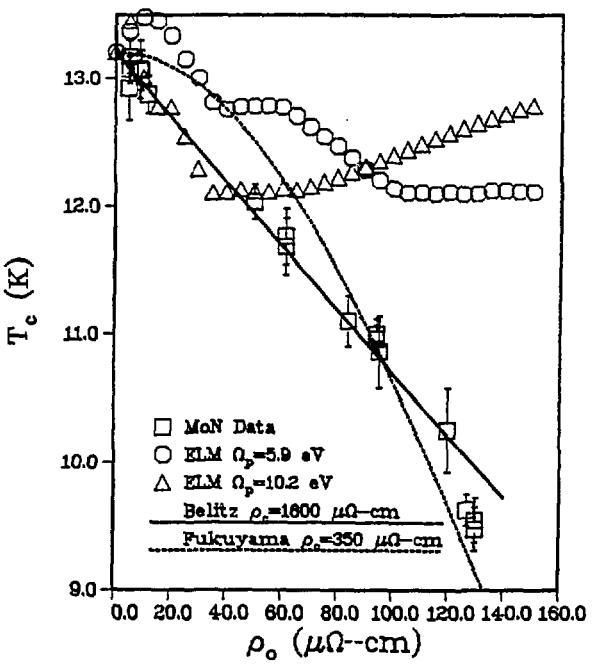

Figure 2. The transition temperature T_c of hexagonal MoN as a function of the residual electrical resistivity ρ_0. Also shown are the theoretical results from the electron lifetime model (ELM), and from the weak localization models of Belitz and of Fukuyama, et al.

To utilize the electron-lifetime model (ELM),[17] N(E) is convoluted with a Lorentzian broadening

function $S(E,E_F,\Gamma)$, that is centered on E_F and has a half width Γ. The energy width Γ is related to the electron lifetime τ and ρ_0 by,[17]

$$\Gamma = \hbar/\tau = 1.34 \times 10^{-4}\ \Omega_p^2\ \rho_0, \qquad (1)$$

where, ρ_0 is in $\mu\Omega$-cm and the energies in eV. The infrared plasma energy Ω_p has not been measured or calculated for hex MoN, but we estimate its value to be within the range 6 to 10 eV, based upon Ω_p = 7 eV for VN,[20] and the observed effective charge carrier densities for other superconducting compounds.[17] Therefore, we have evaluated the effect at both ends of this range, and find the general conclusions unaffected by the detailed value of Ω_p. From the deduced $N(E_F,\rho_0)$, we then calculate $T_c(\rho_0)$ using the Allen-Dynes equation[22]

$$T_c = \frac{\omega_{log}}{1.20}\ \exp\left[-\frac{(1+\lambda_{ep})}{\lambda_{ep} - \mu^*(1+0.62\lambda_{ep})}\right], \qquad (2)$$

and the assumption that the phonons are unaffected, so that $\lambda_{ep}(\rho_0) = \lambda_{ep}(0)(N(E_F,\rho_0)/N(E_F,0))$. We have normalized to the extrapolated $T_c(0)$ = 13.2 K by choosing $\lambda_{ep}(0)$ = 0.8,[20] μ^* = 0.12, and ω_{log} = 323 K. The results plotted in Fig. 2 show the predicted variation in T_c arising from structure in N(E) over a maximum energy breadth ~3 eV about E_F. While there is some detailed sensitivity to the choice of Ω_p, the overall scale of the effect is clearly insufficient to describe the data.

Another recent theoretical approach to the interplay between T_c and disorder involves the application of quantum mechanical corrections to the effects of elastic scattering on the pair propagator and diffusive motion of the quasi-particles.[10,11] These corrections modify the conclusions of Anderson's original theorem,[23] and lead to resistivity-dependent renormalizations that depend on both the strength of the electron-phonon interaction λ_{ep} and of the screened coulomb interaction μ; in particular, they lead to a ρ_0-dependent increase in the coulomb pseudo-potential μ^* appearing in Eq. 2. In contrast to the ELM, in this approach it is assumed there is no change per se in the value of λ_{ep}. In the regime of weak localization, applicable to the disordered compounds and most alloys, one has the conditions $E_F \gg \hbar/\tau \gg \hbar\omega_D$, where ω_D is the Debye frequency. For this case, Fukuyama et al.[11] and Belitz[10] have derived expressions for T_c as a function of ρ_0. Although they have approached the problem from the same viewpoint, different approximations have yielded somewhat different results (see ref. 10).

Curves generated by these models are plotted in Fig. 2. Here, ρ_c is a fit parameter, the magnitude of which characterizes a resistivity scale. According to Belitz,[10] ρ_c is expected to be in the range 1–1.5 mΩ-cm for the present material. The results of ref. 10 are seen to represent the data well, except possibly for the extra observed T_c reduction near saturation. The above considerations indicate that the T_c reductions in hex MoN cannot be ascribed to smearing of a structured density of electronic states, but rather appear to be in good accord with the recent quantum corrections to Cooper pairing and diffusive electronic motion.

Measurements of the upper critical field temperature derivative near T_c should provide values for the renormalized, strong-coupling-weighted density of states $\eta N(E_F)^* = (1+\lambda_{ep})N(E_F)$,[24] where η is the many-body strong-coupling correction for H_{c2} at T_c. It is instructive to compare the apparent $N(E_F)^*$, deduced from the H_{c2} slope, to the band theory predictions for $N(E_F)$ and λ_{ep}, and to check for disorder-dependence in the renormalizing factors given above, and to check for consistency with the assumptions made in the analyses of T_c suppression.

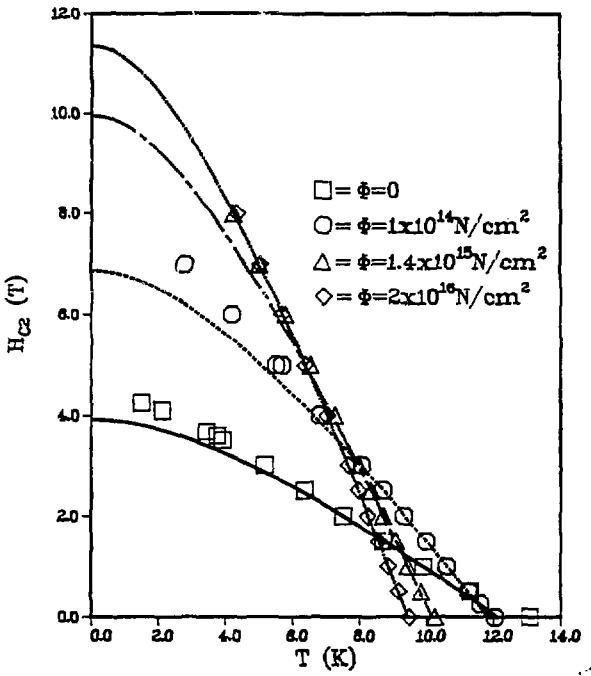

Figure 3. The upper critical field H_{c2} as a function of temperature for some representative N-ion fluences Φ. The curves are the WHH theory in the limit of infinite spin-orbit scattering.

In attempting to do this, we encountered two problems with the behavior of H_{c2} for MoN. The first problem is the inability to obtain a satisfactory fit of the temperature-dependent data to the theory of Werthamer, Helfand, and Hohenberg (WHH),[25] fully accounting for paramagnetic limiting, spin-orbit scattering, and electron-phonon renormalizations. The WHH curves, when matched to the data near T_c, fall below the observations at low temperature, even for the unphysical choice of infinite spin-orbit scattering rate. The deviations are most severe for the as-formed films, and gradually decrease, but do not disappear, with increasing irradiation dose, as seen for some representative data in Fig. 3.

The explanation that immediately comes to mind for this problem is the existence of resistive material inhomogeneities which contribute to an enhanced H_{c2} at low temperature. We believe this explanation is unlikely for the following reasons: i. The diffraction patterns for the best films have sharp peaks and show no second phase. ii. The zero-field transition widths are no broader than for our VN samples, which do not display this problem.[21] iii. The ρ_0 are quite low, whereas the most likely inhomogeneities are substoichiometric MoN_x or Mo_2N, the latter being highly resistive[26] due to the randomly occupied N-sublattice. iv. For some earlier films that had measurable quantities of Mo_2N, the effect on H_{c2} versus temperature was easily detectable as an abrupt increase in slope with decreasing temperature, occuring below about 3 K.

An alternative explanation may lie in the fact that, as far as we know, these are the first determinations of H_{c2} on a hexagonal superconductor with as high a T_c. Judging from earlier measurements of single crystal technetium (T_c = 6.82K),[27] there is likely to be significant H_{c2} anisotropy arising from the band structure. This anisotropy is much larger than, and in addition to, the contribution coming from non-locality, and has been theoretically described.[28,29] Earlier measurements made on high quality, homogeneous bulk polycrystalline technetium showed a low temperature enhancement in H_{c2} over the clean limit WHH theory, of

about the same magnitude (10–15%) that we observe here in hex MoN.[30] Therefore, it seems likely that the H_{c2} anomalies we observe arise from these crystalline anisotropy effects.

The second problem is that $N(E_F)$ found for the as-produced samples is anomalously large, when one uses the expression,

$$\eta \left.\frac{dH_{c2}}{dT}\right)_{T_c} = 3.74 \times 10^{-3}\, N(E_F)^* \rho_0, \qquad (3)$$

where the units of $N(E_F)^*$ are states/eV-MoN-spin, and ρ_0 is in μΩ-cm. This problem can be traced to the inapplicability of the dirty limit to the relatively clean as-formed samples. Using the extrapolated $H_{c2}(0)$ to obtain an upper limit on the BCS coherence length $\xi_0 = 10$ nm, and for reasonable estimates of the transport scattering length ℓ from $\rho\ell = 0.5$ to 1.0×10^{-11} Ω-cm^2, we obtain for the Gorkov parameter[31] $\lambda_{tr} = 0.882\, \xi_0/\ell$ the range of small values 1.0 to 0.3. To deduce $N(E_F)$ from the full $dH_{c2}(T_c)/dT$ analysis[24] requires values for the free area of the Fermi surface, a parameter which presently is unavailable. Therefore, in the following we use only the $N(E_F)^*$ values obtained from application of the dirty limit theory (Eq. (3)) to the ion-irradiated samples.

We plot in Fig. 4 the deduced renormalized density of states $N(E_F)^*$, as a function of the damage resistivity ρ_0. For comparison with the band theory, we again employ the ELM and include in Fig. 4 calculations of the renormalized, smeared density of states $N(E_F)^* = N(E_F,\rho_0)\,(1 + \lambda_{ep}(\rho_0))$, obtained from the theoretical $N(E)$ and $\lambda_{ep}(0) = 0.8$,[20] and for the two choices of Ω_p shown. The experimental results actually fall below those of the ELM, and imply that there are effects of disorder on the electron-phonon interaction λ_{ep} not considered in the ELM, such as a hardening of the phonon spectrum. This observation contradicts one of the assumptions made above in fitting the theories of weak localization to the observed disorder-dependent T_c reductions, namely, that of a constant λ_{ep}. However, at this point, it seems just as likely that the discrepancies in Fig. 4 are due to the application of Eq. 3 to the anomalous H_{c2} data, in view of the potentially large anisotropy effects that are expected to diminish with increasing damage. To clarify this issue, it would be helpful to have heat capacity or tunneling measurements on the materials at various levels of damage, to provide independent information about $N(E)$ and the phonon structure, or band theoretical evaluations of the Fermi surface averages needed to estimate the effects of H_{c2} anisotropy.

Finally, we briefly remark on the x-ray diffraction analyses of the irradiated films. Over the range of ion-fluences extending up to $\Phi = 2 \times 10^{16}$ N-ions/cm^2, corresponding to a maximum damage energy deposition $E_a = 200$ eV/atom, the lattice parameters were found to deviate only slightly from those of the as-produced MoN, showing a slight, uniform reduction in the cell dimensions of about 0.5 ± 0.1%, recorded near the saturation dose. This is in contrast to the response to radiation-damage of A15 materials which undergo a lattice expansion,[32] and of B1-structure NbN damaged with Ar ions, which shows first a lattice expansion followed by a contraction at high fluences.[33] The present observations are in accord, however, with those in MoC irradiated with He,[34] and with our recent measurements on N-ion irradiated VN.[14]

Summary and Conclusions

High quality, essentially single phase thin films of hexagonal structure MoN were fabricated and irradiated with nitrogen ions at energies designed to produce a uniform damage deposition profile, and up to fluences corresponding to several atomic displacements/lattice site. The effects of the irradiation-induced disorder on the lattice structure, ρ_0, T_c, and $H_{c2}(T)$ were compared to some benchmark materials, and assessed in terms of the current theoretical ideas of disorder effects on the electronic structure and superconducting properties.

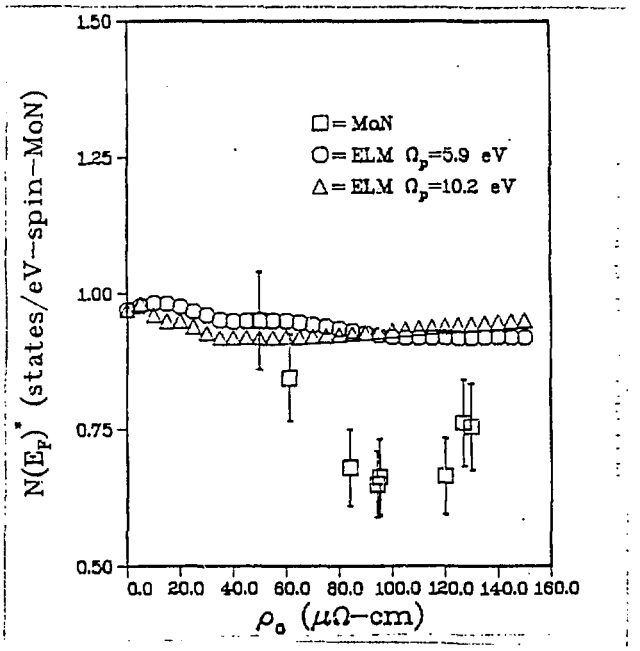

Figure 4. The renormalized electronic density of states $N(E_F)^*$ as a function of residual resistivity ρ_0. For comparison, the ELM, applied to the band theoretical results, is included for two extreme values of the infrared plasma frequency Ω_p.

Briefly, we find:

(1) The disorder-induced degradation of T_c is much smaller for hexagonal MoN than for the high-T_c A15-structured Nb_3Sn,[32] or for the B1 materials VN,[14] with total reductions in T_c at saturation of 27%, 84%, and 42%, respectively.

(2) The reduction in T_c is nearly linearly-related to the damage resistivity ρ_0. An analysis employing the electron lifetime model (ELM) shows that this behavior cannot be related to a simple smearing of the density of states near the Fermi level, $N(E_F)$. The data appear to be more accurately described by the recent theories of weak localization, that lead to ρ_0-dependent renormalization enhancements in the coulomb psuedopotential μ^*.[10,11]

(3) With increasing lattice disorder in hexagonal MoN, the observed upper critical field $H_{c2}(0)$ monotonically increases but has a temperature dependence that cannot be adequately described by the full, renormalized WHH theory.[24] We deduce a probable explanation for this discrepancy lies in the strong anisotropy arising from the uniaxial crystal structure.[28,29] In addition, the diminution of this anisotropy with increased disorder may account for an apparent reduction in the electron-phonon interaction parameter λ_{ep}, found by applying dirty limit analysis to the H_{c2} temperature derivative near T_c.

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