Muon spin relaxation in Re-substituted $HgA_2Ca_{n-1}Cu_nO_{2n+2+x}$ (A=Sr,Ba; n=2,3) superconductors

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The muon relaxation rate σ for the Re-substituted $HgSr_2CaCu_2O_{6+y}$ and $HgBa_2Ca_2Cu_3O_{8+y}$ superconductors has been measured as a function of temperature. The low-temperature values of σ fall close to the universal Uemura line $T_c(\sigma)$, as observed for other layered superconductors; therefore our data show no evidence of a reduced anisotropy, i.e., interlayer metallization, induced by Re. The in-plane penetration depth values obtained for these 1212 and 1223 compounds are found to be significantly different ($\lambda = 1665$ Å and $\lambda = 1310$ Å, respectively). The roles of the inserted CuO_2 planes, the Ba/Sr substitution and the character of the spacing layers are considered to discuss the observed features. [S0163-1829(99)00634-7]

INTRODUCTION

One of the fundamental features of the superconducting cuprates is their layered structure. They are made of superconducting CuO_2 planes—or blocks of planes—separated by other layers which act as charge reservoirs and, depending on their atomic constituents and thickness, can be insulating, metallic, or even superconducting. This anisotropic structure is thought to be at the origin of most of the unusual superconducting properties of these compounds, as, for instance, the high critical temperature T_c and the low irreversibility line.

Among the high-T_c superconductors, Hg-based cuprates with general formula $HgA_2Ca_{n-1}Cu_nO_{2n+2+x}$ (A=Sr,Ba) deserve special attention.[1] They can be synthesized with different number ($n \leqslant 6$) of Cu-O layers per superconducting block; and the $n=3$ member of this family (with A=Ba) displays the highest T_c ever reported, 134 K, which can be increased up to 164 K by applying hydrostatic pressure. Several partial cation substitutions have been tried in order to (i) further raise T_c and (ii) stabilize these compounds of difficult synthesis. Most of the investigated compositions are detrimental to T_c and do not result in a stabilization of the compounds.[2] The remarkable exception is the partial substitution of Hg by Re, which has been demonstrated not to lower the maximal T_c and stabilize these phases.[3] Additionally, the $Hg_{1-x}Re_xSr_2CaCu_2O_{6+x}$ compound has been reported to display a 50-fold increase of the irreversibility line, with regard to the undoped $HgBa_2CaCu_2O_{6+x}$.[4,5] Taking into account the shrinkage of the crystal lattice induced by Re and the high valence of this element, this effect has been interpreted as due to a reduction of the superconductor anisotropy, caused by the metallization of the spacing layers between the superconducting CuO_2 planes.[5] In fact, recent magnetization measurements on grain aligned $Hg_{1-x}Re_xBa_2Ca_2Cu_3O_{8+x}$ have allowed us to estimate an anisotropy ratio $\gamma \equiv (m_c/m_{ab})^{1/2} \geqslant 3-8$ from the slopes of the high-field reversible magnetization;[6,7] these values are much lower than $\gamma \approx 52$ obtained from angular dependent magnetization in single crystals of unsubstituted $HgBa_2Ca_2Cu_3O_{8+x}$.[8] However, no direct evidences of a modification of the charge-carrier density or induced interlayer metallicity by Re have been obtained, except for early reports of a high value of the ratio between the superfluid density n_s and the effective mass m^* for $Hg_{1-x}Re_xBa_2CuO_{4+x}$ ($T_c \approx 90$ K) and $Hg_{0.75}Re_{0.25}Sr_2CaCu_2O_{6+x}$ ($T_c < 90$ K) samples, as obtained from muon spin relaxation measurements.[5] Disclosing the role of interlayer metallization may open new strategies for developing useful materials, and to understand the interlayer coupling and its role in high-temperature superconductivity.[9]

The muon spin-relaxation (μSR) technique can indeed be very useful to get an insight into the doping level and superconducting dimensionality. The muon depolarization rate σ reflects the field distribution inside the sample, which for a superconductor in the mixed state is determined by the penetration depth λ. In the London model, λ depends basically on the ratio between the effective mass m^* and the superfluid density n_s, so that one has $\sigma \sim \lambda^{-2} \sim n_s/m^*$. Uemura *et al.*[10] made the remarkable observation that for most underdoped and close to optimally doped cuprates, the $T_c(\sigma(T=0))$ data fall on a unique line (the so-called Uemura line); deviations from this line occur for overdoped samples,[11] i.e., when T_c is lowered for increasing n/m^*, and also for cuprates with chains, which show a saturation of T_c.[12,5] It is therefore believed that the Uemura line reflects the behavior of the superconducting Cu-O planes; low doped cuprates falling on it would have basically a two-dimensional (2D) character, whereas the least anisotropic ones, with metallic or superconducting interlayers, would deviate from the line.[5]

The observation of a universal $T_c(\sigma)$ dependence has stimulated important discussions which go deep into the nature of the pairing mechanism.[10,5] At this moment no clear theoretical consensus has been reached, but as a matter of fact it is important to explore $T_c(\sigma)$ for new superconducting systems. As a consequence of Uemura's empirical law, com-

parison of the σ values for Hg-based cuprates with and without Re is of major interest to see whether the substitution has altered the n_s/m^* ratio; and the position of the $T_c(\sigma(T=0))$ values with regard to the Uemura line should in turn provide information on the possible metallization of the interlayers. We note in passing that determinations of the anisotropic penetration depth from data taken on grain aligned samples have the drawback of being extremely sensitive to sample quality (superconducting fraction) and orientation, and crystallite shape effects. To this respect, μSR appears as a most suitable alternative technique to explore anisotropy modifications.

In this paper we report on muon spin-relaxation measurements on several Re-substituted samples, having compositions $Hg_{0.82}Re_{0.18}Sr_2CaCu_2O_{6+x}$ and $Hg_{0.82}Re_{0.18}Ba_2Ca_2Cu_3O_{8+x}$, close to optimal doping. This is an important issue, as previously reported data on Re-1212 were obtained on overdoped samples.[5] The obtained $T_c(\sigma(T=0))$ data for our samples ($n=2$ and $n=3$) fall very close to the Uemura line. This observation is of significance because it provides indications that Re-substitution in these Hg-based phases does not substantially modify the anisotropy; that is, our μSR data do not evidence any Re-induced interlayer metallization, in contrast to the earlier result of Ref. 5. We discuss this discrepancy and argue possible reasons for the enhancement of the irreversibility line earlier observed.

EXPERIMENTAL

The measured samples were as-grown powders of composition $Hg_{0.82}Re_{0.18}Ba_2Ca_2Cu_3O_{8+x}$ and $Hg_{0.82}Re_{0.18}Sr_2CaCu_2O_{6+x}$, prepared by the sealed quartz tube method.[13] In the following, they will be refered to as the Ba3 and Sr2 samples, respectively. The Rietveld refinements of the powder x-ray diffractograms revealed that they are almost single phased, with only minor traces ($<10\%$) of $HgCaO_2$ and lower n phases. The refined c and a cell parameters agreed well with those reported for Re-substituted Hg-based cuprates.[3,14,15]

The critical temperatures of the two studied samples, obtained from the diamagnetism onset of the low-field dc magnetization (Fig. 1), are $T_c=132.5$ K and $T_c=107.5$ K, respectively for Ba3 and Sr2. These high values imply that they are close to optimally doped. In fact, the T_c value for the $n=3$ sample is only slightly below that of the unsubstituted compound ($T_c=135$ K, Ref. 1). The $n=2$ sample with Sr displays the highest T_c reported so far for this Re-substituted phase;[15] we note that the unsubstituted phase ($n=2$, $A=$Sr) has not been synthesized so far.

The Meissner susceptibility $\chi_{\text{Meiss}}\equiv|dM/dH|$ obtained from the low-field/low-temperature magnetization is lower than 1, therefore implying that the magnetic field is not totally screened from the sample volume; from the χ_{Meiss} values on extracts that the full screened volumes are $x_{\text{SC}}=77\%$ and $x_{\text{SC}}=50\%$, for the Sr2 and Ba3 samples, respectively. As discussed in a previous paper,[16] this result appears related—at least partially—to the shape of the superconducting grains, which are tiny platelets of thickness <0.5 μm, smaller than the estimated penetration depth along the c axis ($\lambda_c\approx 6$ μm for the $n=3$ phase, Ref. 17).

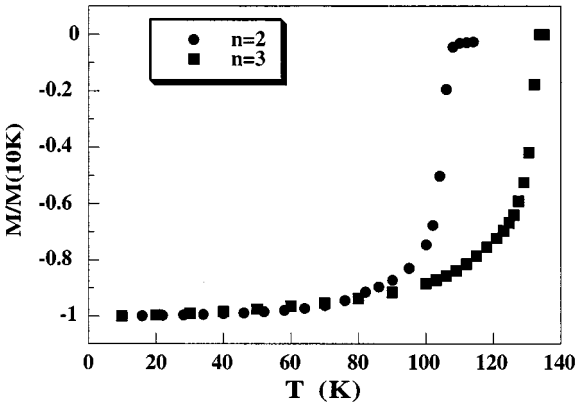

FIG. 1. Low-field dc magnetization of the two powders measured: Sr2 and Ba3. From the low-temperature magnetization values, superconducting volume fractions of 83 and 55% are obtained, respectively.

Transverse field μSR experiments were performed at ISIS (Rutherford-Appleton Laboratory, United Kingdom). The powders were cooled in a closed-cycle cryostat, after applying a field H ranging between 100 and 700 Oe. Higher fields could not be used because of the reduction in the measured amplitude assymmetry at high precession frequencies. The muon relaxation rate was measured at temperatures between 10 K and above T_c.

RESULTS

Figure 2(a) displays the time-dependent μSR amplitude assymmetry for sample Ba3 at $T=125$ K. The fast amplitude decay observed at short times is due to the presence of vortices inside the material. As it is well known, a nonuniform magnetic-flux distribution leads to a fast depolarization of the muons and thus to a decay of the measured μSR amplitude. At longer times, a slow decaying signal with roughly the same periodicity and $\sigma_n\approx 0.1\,\mu s^{-1}$ becomes apparent. This signal is also observed above T_c [Fig. 2(b)] and is hence not related to the superconducting state. It has a value similar to the contribution earlier adscribed to Cu nuclear dipole moments,[18,19] and thus it might arise from them. The coexistence of the superconducting and this background contribution below T_c implies that a fraction of the sample does not display the modulated field distribution associated to an Abrikosov flux lattice. As discussed in the preceding section, this behavior may arise from the crystallites aligned parallel to the applied field, or from the contribution of the minor impurity phases present in the samples. The superconducting contribution, though, is distinct enough and clearly dominates at short times.

In order to obtain the muon relaxation rate associated to the superconducting state, $\sigma\propto\lambda^{-2}$, the recorded precession signal was fitted to a two-component function; the first component—a Gaussian, $\exp(-\sigma^2t^2/2)$—accounts for the decay due to the field modulation inside the superconductor; the second—minor—component accounts for the slow decaying background contribution, observed also in the normal state. The obtained fits and σ values were verified not to depend on the muon beam size, nor on the precise time dependence of the background contribution.

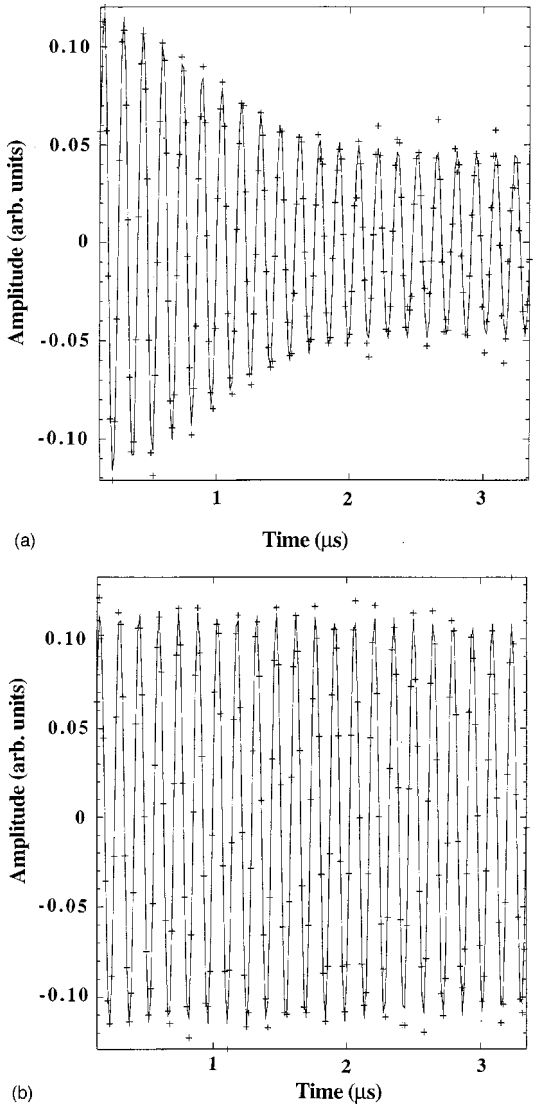

FIG. 2. Time-resolved μSR signal, measured for the $n=3$ sample at 500 Oe for $T=125$ K (a) and 140 K (b).

As mentioned above, the muon depolarization is caused by the nonuniform flux distribution inside the sample. In the mixed state, the Abrikosov vortex lattice produces a modulated field distribution with characteristic length scale λ. Additional flux gradients associated to pinning may also contribute to σ, thus making hard the extraction of λ. For those reasons, field cooled processes are required in order to stablish a homogeneous flux distribution inside the superconductor, and, additionally, an intervortex separation a_0 shorter than λ is needed to extract reliable λ values.

The dependence of the relaxation rate on the applied magnetic field H after field cooling was measured for a temperature $T \ll T_c$ (Fig. 3). As expected, σ increases with H at low fields, when a_0 is larger than λ. At high enough fields, when $a_0 \approx \lambda$, σ must saturate; accordingly, the $\sigma(H)$ dependence in Fig. 3 shows a slowing down at the highest available fields, although not a clear saturation is attained. Nevertheless, the increase of σ with field for $H \approx 500$ Oe is only slightly above the error bars. Therefore the obtained relaxation rate values are expected to be close to the actual ones, somewhat underestimating them.

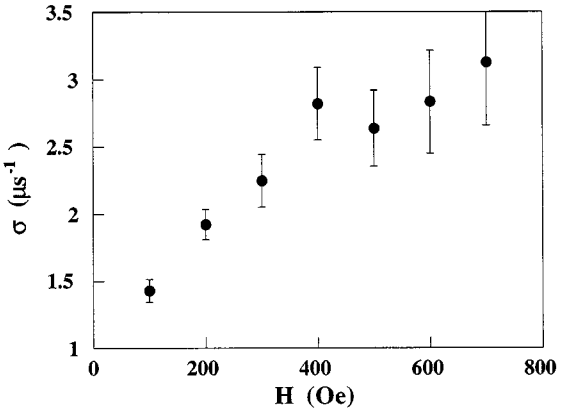

FIG. 3. Field dependence of the μSR depolarization rate σ of sample Sr2 at 20 K.

The evolution of σ with temperature was thus measured using a field of 500 Oe. The obtained values for both samples are shown in Fig. 4. They display the behavior usually expected for $\lambda^{-2} \sim 1-(T/T_c)^n$: a flat temperature dependence far from T_c and a sharp reduction when approaching T_c. The saturation—low temperature—values of the relaxation rate are $\sigma = 2.5 \pm 0.3\ \mu s^{-1}$ and $\sigma = 4.1 \pm 0.4\ \mu s^{-1}$, for $n=2$ and $n=3$, respectively. From these values of σ and using the $\lambda[nm] = (7.08610^4/\sigma[\mu s^{-1}])^{1/2}$ (Ref. 12) relationship, an estimate of the effective zero-temperature penetration depth can be obtained. We obtain,

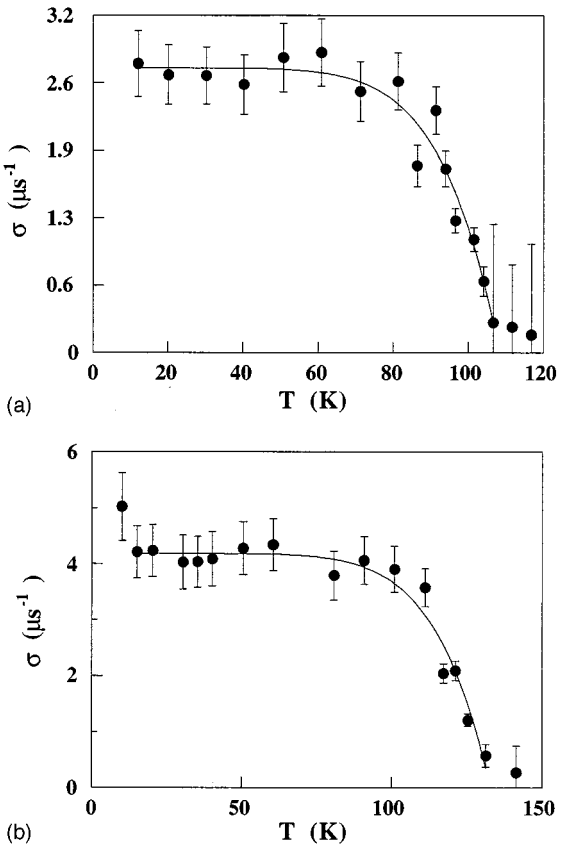

FIG. 4. Temperature dependence of the the μSR depolarization rate σ for samples $n=2$ (a) and $n=3$ (b) recorded at $H=500$ Oe. The solid lines are fits to a law $[1-(T/T_c)^n]$, as discussed in text.

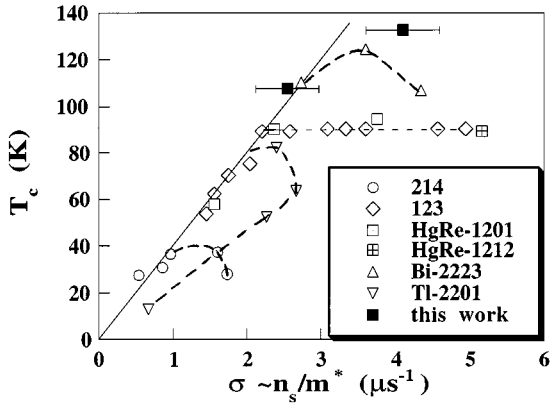

FIG. 5. T_c versus $\sigma(T=0)$ (Uemura plot) for several cuprate superconductors with different structures and doping levels. The lines through the points are guides to the eye. Full squares are this work's data. The other symbols are data extracted from Refs. 5 and 11. Note that under and close to optimally doped cuprates follow a unique $T_c \sim \sigma$ line; deviations to the right occur for overdoped compounds (see dashed lines through the Tl-2201 and some 124 and Bi-2223 data points) and cuprates with metallic spacing layers (Y-123 and Y-247).

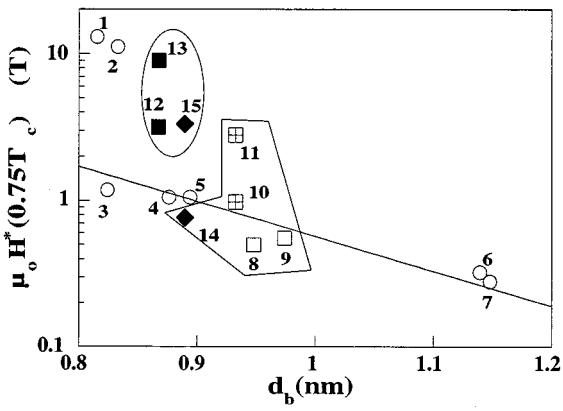

FIG. 6. Irreversibility field $H^*(T=0.75T_c)$ as a function of the interlayer spacing d_b for several cuprate superconductors, listed in Table I. Note that diamonds are this work's data. The other symbols are data extracted from Refs. 4, 5 and 15. Squares represent several Hg-based superconductors: open squares are undoped samples containing Ba, crossed squares are Re-doped samples with Ba, and full squares are Re-doped samples with Sr. Open circles are other HTS data. In order to make clearer the plot, we have enclosed the data from Hg-based materials with Ba in a polygon, and those on Hg-based with Sr in an ellipse. The solid line describes the general behavior considered typical of cuprates without Cu-O chains (Ref. 5).

respectively, $\lambda = 1665\,\text{Å} \pm 90\,\text{Å}$ and $\lambda = 1310\,\text{Å} \pm 70\,\text{Å}$ for the $n=2$ and $n=3$ samples. For a polycrystalline uniaxial material, the observed depolarization rate σ is weighted by both λ_{ab} and λ_c. However, it has been shown that if the anisotropy ratio λ_c/λ_{ab} of the superconductor is higher than 5—as expected for Hg-based compounds—the effective λ mainly reflects the in-plane penetration depth λ_{ab}. Therefore the above obtained values can be considered good estimates of λ_{ab} for our two samples.

In Fig. 5 we plotted the σ values obtained in the present study, together with the $T_c(\sigma)$ data points corresponding to several cuprates with different structure and doping level. As observed by different authors,[10,5] the $T_c(\sigma)$ of most cuprate superconductors under and close to optimally doped fall onto a unique line—the so-called Uemura line—thought to reflect the behavior of the superconducting Cu-O planes. Deviations from this line have been observed for overdoped samples,[11] which reveal a decrease of T_c as σ increases, and also for compounds with Cu-O chains. In the latter case, the saturation of T_c for increasing σ values has been associated to the metallic character of the interlayers, and therefore to a reduced anisotropy.[5]

It may be appreciated that our σ values lie very close to the Uemura line. This result is in sharp contrast with the previous μSR data reported for two Re-doped superconductors by Tallon *et al.*[5] As reflected in Fig. 5, these authors reported quite high σ values for a Hg-1212 sample with 25% Re, and for another Re-doped Hg-1201 sample. These high values were taken by the authors as an evidence of the metallization of the interlayers, induced by rhenium. Such a metallization should result in 3D superconductivity, which would reflect in a deviation of $T_c(\sigma)$ from the Uemura line, as observed for other high-temperature superconductor (HTS) with Cu-O chains.[5,12] Our data for both the $n=2$ and $n=3$ close to optimally doped samples do not reveal any substantial enhancement of σ with Re substitution. We note that the lower T_c of the 1212 sample in Ref. 5, in comparison with $T_c = 107.5\,\text{K}$ of our Sr2 sample implies that the former is not optimally doped; overdoping could account for the reported deviation from the Uemura line, as observed for instance in Tl-2201.[11] The $\sigma \approx 4\ \mu\text{s}^{-1}$ value reported for the (Hg,Re)-1201 sample, having a $T_c = 90\,\text{K}$ and thus close to optimal doping,[5] remains puzzling.

DISCUSSION

The main conclusion from the data reported above is that optimally doped Re-substituted Hg-1212 and Hg-1223 superconductors follow the universal behavior of Uemura, regardless of whether they contain Ba or Sr. Thus no clear evidences for interlayer metalization have been obtained.

The absence of interlayer metallization by Re revealed by our data is in fact consistent with the unalterated maximum T_c value. One could expect that any reduction of the effective distance between superconducting Cu-O blocks, induced by Re, may modify T_c, either by changing the charge transfer or by proximity effect. The absence of any measurable effect of Re on T_c appears to indicate then that the shrinkage of the unit cell and oxygen uptake produced by this substitution might be enough to stabilize the phases, but not to sensibly modify the intrinsic superconducting properties (namely, T_c) of this family of compounds.

Another important feature is the observed difference between the λ (or σ) values for the Sr2 and Ba3 samples. This variation, larger than the error bars associated, implies that the ratio n_s/m^* increases with n, or upon substituting Sr by Ba. The currently available λ data for unsubstituted Hg-based superconductors with different n, extracted from magnetization measurements, do not show any clear tendency of systematic variation of λ with n.[17,20,21] Therefore the distinct λ (or σ) observed for Sr2 and Ba3 in our data, could be

TABLE I. Correspondence between the labels and compounds depicted in Fig. 6.

Label	Sample	Reference
1	Y-123	5
2	Y-247	5
3	Y, Ca-123	5
4	Tl-1223	5
5	Tl-1212	5
6	Tl-2223	5
7	Tl-2212	5
8	HgBa-1223	5
9	HgBa-1212	5
10	(Hg,Re)Ba-1223	4
11	O_2 annealed (Hg,Re)Ba-1223	4
12	(Hg,Re)Sr-1223	4
13	(Hg,Re)Sr-1212	3
14	(Hg,Re)Ba-1223	this work
15	(Hg,Re)Sr-1212	this work

rather related to a reduction of the charge transfer, due to the shortening of the *c*-axis parameter when substituting Ba by Sr. Values of λ for other Hg cuprates containing Sr have not been reported, to our knowledge. This is, though, an important point to verify, as we shall see, since the shrinkage of the unit cell due to this cation substitution is of the same order of magnitude as that induced by the partial substitution by Re.[15]

Now, an important question arising is why the irreversibility line $H^*(T)$ of Re-doped compounds is increased, if the strong anisotropy of Hg superconductors is not sensibly modified. First of all, we note that the most significant Re-induced enhancement of $H^*(T)$ was reported for a $n=2$ sample with Sr, which exhibited a 50-fold increase with regard to that of an unsubstituted $n=2$ sample with Ba.[4] The rise of the irreversibility lines for other Re-substituted compounds—namely, those with Ba instead of Sr—is much less evident;[14,22] in any case, the position of $H^*(T)$ appears strongly dependent on the oxygen content of the sample.[4,15] Therefore the irreversibility line appears quite related to the detailed microstructure of the samples, as it should be expected, but evidences of the unique effect of Re are lacking.

In any event, it is interesting to stablish how the irreversibility line behaves for the different Hg-based cuprates. In Fig. 6 we plot $H^*(T=0.75T_c)$, commonly used to characterize the irreversibility line, as a function of the distance d_b between superconducting blocks in several as-grown Hg-based and other cuprates. The materials which have higher H^* than the usually obtained for a given d_b distance are in fact the Re-substituted Hg-1212 samples with Sr. All the Hg compounds with Ba lie on the basic $H^*(d_b)$ line, regardless they have or do not have Re; the only exception is a O_2 annealed (Hg,Re)-1223 sample with Ba reported in Ref. 4. It appears thus that the presence of Sr might be at least as relevant as Re for the enhanced irreversibility observed. Measurements of $H^*(T)$ on undoped ceramics of Hg-1212 and Hg-1223 with Sr should provide an experimental way to discriminate between the role of Sr and Re. In fact, recent data on $Hg(Ba_{1-x}Sr_x)_2CaCu_2O_y$ materials with different Ba/Sr ratio indicate an enhancement of pinning for increasing Sr content, in spite of the accompanying T_c decrease.[23]

In summary, the present μSR data on Re-substituted $n=2$ and $n=3$ Hg-based superconductors, lead to $T_c(\sigma)$ values that fall onto the universal Uemura line; therefore they do not provide any indication of Re-induced reduced anisotropy metallization of the interlayers, as earlier suggested. On the other hand, analyses of our data and that from literature allow to suggest a relevant role of the lattice contraction when substituting Ba by Sr, both on the observed σ (and thus on n_s/m^*) and the irreversibility line. Detailed analyses of the structural and superconducting properties of the $n=2$ and $n=3$ with different Ba/Sr contents are needed to definitely settle the importance of interlayer coupling in these compounds.

ACKNOWLEDGMENTS

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