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Effect of spacer layer on flux-pinning properties of iron-based superconductors

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Abstract: We investigated the magnetic flux-pinning in iron-based 1111-type (REFeAsO, RE= rare earth) and 11-type ($FeSe_{1-x}Te_x$) superconductors by third harmonic ac-magnetic susceptibility measurements, a technique sensitive to the dynamics of magnetization, probing non-linear processes in flux pinning transport. Despite the larger thermal fluctuations due to the high critical temperature and large anisotropy, flux dynamics of 1111-type pnictides points out a more efficient pinning mechanism than the one in the 11-type chalcogenides. We have associated the stronger pinning in the 1111-family to the presence of REO (RE=rare earth) spacer layers separating the active FeAs-layers unlike the 11-chalcogenides in which no spacer layers are present. Therefore, the disorder in the FeAs layers induced by the misfit strain due to spacer layers has an important role in the enhanced flux pinning of 1111-type superconductors.

Keywords: Fe-based superconductors, pnictides, chalcogenides, vortex-dynamics; flux-pinning, a.c. susceptibility, space layer

1 Introduction

The discovery of superconductivity in $LaFeAsO_{1-x}F_x$ in 2008 [1] has triggered a large number of experimental and theoretical studies to explore layered iron-based compounds [2–5], resulting in the synthesis of many new superconducting materials with differing transition temperatures. These new superconductors are characterized by several chemical compositions. Among the many stoichiometric compounds, we have the REFeAsO (RE = rare earth) materials that belong to the 1111 family, i.e., compounds with four elements with a 1:1:1:1 ratio. Other main families are the 122 compounds, e.g., the $BaFe_2As_2$ [6], the 111 compounds, e.g., the LiFeAs [7] and the 11 compounds, e.g., the FeSe [8].

Among iron-based superconductors, the 1111 family holds the record of highest critical temperature, as high as 55 K [9, 10]. All the iron-based superconductors share a common chemical element, i.e., the FeX layer (X = As, P, S, Se, or Te). The FeX layers consist of a square array of Fe sandwiched between two layers made by X atoms with a tetrahedral coordination between the Fe and X anions. These layers are either stacked one over the other (e.g., in the 11 family), or separated by "spacer" layers with different chemical configurations (e.g., REO in the 1111 family, or alkaline atoms in the 122 family). While the "spacer" layers are known to stabilise the average structure, the fundamental electronic structure of these materials is mainly driven by the Fe 3d states, hybridized with the electronic orbitals of the X anions [11] and characterized by highly covalent Fe-X bond lengths [12].

Since both superconductivity and itinerant striped magnetism occur in the FeX layers, the role of the "spacers" has been one of the highly debated questions in these materials. The misfit strain between the spacer layers on the FeAs layers occurs [13] inducing an anisotropic internal pressure on the FeX layers which together with a proximity to a Lifshitz transition driven by a new appearing Fe 3d small Fermi surface pocket [14, 15] gives a particular inhomogeneity giving nanoscale phase separation like in cuprates [15–19] which has been investigated

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by XANES [20–22] and predicted for Lifshitz transition in multiband Hubbard model [23, 24]. From the application point of view, it is very important to explore this complex disorder controlled by space layers in the superconducting properties, such as the flux-pinning as it was made recently in cuprates [25].

The anisotropic internal pressure on the FeX layers is dependent on the ionic radius size of the RE. This effect, in turn, leads to a flux pinning type efficient on the whole FeAs plane superconductor with the increase of the disorder [21, 22]. This effect cannot be present in superconductors 11 Fe-HTSC since there is not REO layer in these compounds. Furthermore, a comparison of the crystal structure 1111 Fe-HTS and 11 FeSe HTSC points out this aspect (see fig. 2, pag. 3, in Ref. [5]).

However, in all the Fe-based HTSC families will be present a second flux pinning type statistically distributed and connected to the local disorder induced by doping F, in 1111 Fe-HTSC family [26] and by composition changing between Se and Te atoms in the 11 Fe-HTSC family [27, 28].

These pinning type will have effects on a smaller scale than the coherence length [29] also considering the coherence length ξ in these Fe-based HTSC is about 2 nm [30] and on a larger scale because the local distortions will be autocorrelated macroscopically for the whole sample [18, 25, 29].

From the general point of view, to know, to control, amplify, the flux pinning is of great technological importance, in fact, determines the critical current in superconducting [29]. The flux pinning arises from complex local cooperative situations: defects, stress, dislocations, precipitated, doping, etc contribute statistically to realize flux pinning both weak and strong [29, 31]. Also in the HTSC, the dimensionality, the chemical structure, have a role which further complicates and modifies the flux pinning. The temperature and magnetic field realize different scenarious where the pinning statistical sums and FLL can have different configurations such as, single vortex or small/medium/large flux bundles [32]. Additional and important effects are due if the material is in polycrystalline form where the grain boundary causing weak flux pinnings which limits the critical current value [33]. These issues have been extensively discussed in the Fe-based superconductors [26, 27, 34].

In this work we have addressed this problem and present a comparative study of magnetic flux-pinning properties of iron-based superconductors with and without "spacer" layers. In particular, we have measured the third harmonic of the magnetic ac-susceptibility of 1111 family ($NdFeAsO_{0.86}F_{0.14}$ and $SmFeAsO_{0.85}F_{0.15}$) characterized by the REO spacer layers [35–38] and the 11 family ($FeSe_{1-x}Te_x$), containing only the stacking of FeX active layers without any "spacer" layers. Multi-harmonic ac-susceptibility is a powerful tool to probe flux-pinning properties [39]. In fact, the high harmonic coefficients, χ_n, are very sensitive to the non-linear flux-pinning interaction in the superconducting phases [40–44].

2 Experimental details

All samples are polycrystalline. Details of the synthesis process and characterization can be found for $NdO_{0.86}F_{0.14}FeAs$ sample in reference [45], for $SmO_{0.85}F_{0.15}FeAs$ sample in reference [46], for the $FeSe_{0.88}$, $FeSe_{0.5}Te_{0.5}$ and $FeSe_{0.25}Te_{0.75}$ samples samples in references [28, 47]. To perform analysis on flux pinning dominant processes in single crystals present in the sample we use the analysis of the higher harmonic (III Harmonic) behaviour of the multi-harmonic ac susceptibility in function of temperature and magnetic field. In fact, the high harmonic components ac detect only the non-linear components in the signal, separate and amplify the signals due to different superconducting phases present in the sample [41–44, 48–51], even when there are small amount of superconducting phases or evanescent superconducting phases are present [52, 53]. The latter are connected to changes of the effective flux-diffusivity tuned by the flux-pinning interaction [39, 40].

In particular, the signal that will show the highest critical temperature will be connected to the superconducting behavior of separated single crystals present in the polycrystalline sample [49]. In this paper we will account only this behaviour that indicates the average superconducting behavior of single crystals distributed uniformly in polycrystalline samples.

To deduce from the multi harmonic ac susceptibility measures the quality of interaction between 'Flux Line Lattice' (FLL) and the pinning phenomena present in the sample is sufficient to have the measures in arbitrary units and compare them with each other. The ac susceptibility in this paper has the following arbitrary units: (V/Hz/Tesla/gr). Underline that the magnetic measurements are dependent on the volume and weight of the sample then the comparison between the different samples is achieved considering the density of the sample. The measured signals will be referred to the weight of each sample (gr) and the volumetric factor called 'fill factor' which takes into account the ratio between the volume of the pick-up coil with respect to the sample volume. The flux pinning quality will be determined by the temperature width of the module ac susceptibility third harmonic signal and the value of its amplitude. Since there are homogeneous distribution of single cristals in the samples the demagnetization geometric factors [37] will be similar in all samples and the signal differences are attributable only to the intrinsic structure of the grains.

The AC multi-harmonic susceptibility measurements were performed in the LAMPS laboratory at the *Laboratori Nazionali di Frascati* of the INFN, using a gradiometer based on a bridge of two pick-up coils cooled in a thermally controlled He gas-flow cryostat, where a superconducting magnet operates up to 8 T. All measurements have been performed under a zero field cooling (ZFC) set-up, i.e., the sample has been slowly cooled to 4.4 K without applying a magnetic field, then an AC magnetic field of 9.8 Gauss at 1070 Hz has been turned on. AC susceptibility multi-harmonic measurements have been performed during the heating process. In particular, to investigate the role of the spacer layers on the flux-pinning of these systems, we have focused to the third-harmonic susceptibility which has the highest amplitude respect to the other high harmonic components. Signals have been normalized to the sample weight and scaled with the sample fill factor (FF), i.e., the ratio between the volume of the sample and of the pick-up coil. The FF values of the investigated samples are shown in Table 1.

3 Results and discussion

Figure 1a shows the first harmonic of the ac-susceptibility measured as a function of temperature on the superconducting $NdFeAsO_{0.86}F_{0.14}$ system containing a spacer layer and the $FeSe_{0.88}$ sample with no spacer layer. The figure shows a clear and sharp diamagnetic transition in both samples across the superconducting transition temperature (T_c (onset)), estimated to be 48 K and 8 K, respectively. Fig. 1b showing the AC-susceptibility signal as a function of the renormalized temperature scale ($t=T/T_c$) points out clear differences in the magnetic response of these two samples. Indeed, while the diamagnetic signal in the 1111 system is sharply saturating to a minimum value, the signal of the 11 is still changing down to 0.6 T_c, and its value remains substantially higher than the 1111 counter part.

To have a further insight to the magnetic characteristics, we have analysed the third harmonic of the AC-susceptibility on both systems. Fig. 2a shows the temperature dependence of the third harmonic $|\chi_3|$ at $H_{dc} = 0$ T for both $NdFeAsO_{0.86}F_{0.14}$ and $FeSe_{0.88}$ samples. The former shows just an intense and single sharp peak, pointing out to an efficient pinning inside the materials [40], while the latter is characterized by a broad and asymmetric distribution related to a different flux pinning mechanism. This broad peak suggests the presence of only an intra-grain phase. According to the critical state model [41, 42], the observed profile of the susceptibility peak in the $NdFeAsO_{0.86}F_{0.14}$ system is a characteristic feature of a stronger magnetic flux-pinning.

To characterize the pinning properties, we have compared in Fig. 2b the Cole-Cole plots, i.e., a plot of χ''_3 vs. χ'_3, of the two samples under the same magnetic field and frequency. As the reference, represented by the dashed curve, we have also plotted the Bean's model independent of the frequency, [54, 55] corresponding to a homogeneous bulk pinning 'lens' shape [56]. The curves reveal a similar bulk flux-pinning interaction for both $NdFeAsO_{0.86}F_{0.14}$ and $FeSe_{0.88}$ samples although the area of the former is larger. The observed behaviour rules out the possibility of a contribution due to surface barrier pinning that is on the contrary characterized by a 'cardiod' Cole-Cole χ_3 plot [57]. The shape of the Cole-Cole plot for the $NdFeAsO_{0.86}F_{0.14}$ is the closest to the Bean's model curve, indicating poorer frequency dependence and a stronger pinning force, compatible to a higher critical current for this system [39].

We have also studied another member of the 1111 family, i.e., the $SmFeAsO_{0.85}F_{0.15}$, characterized by the highest superconducting transition temperature among the iron-based superconductors. The comparison in Fig. 3a of the third harmonic of the AC-susceptibility as a function of the reduced temperature T/T_c for the two 1111 systems shows a different intra-grain connectivity between them. However, both show a sharp third harmonic peak, albeit the one for the $NdFeAsO_{0.86}F_{0.14}$ system is much broader. Differences in the flux pinning properties can be analysed looking at the Cole-Cole plots of the two systems shown in Fig. 3b where the $SmFeAsO_{0.85}F_{0.15}$ behaviour is in reasonable agreement with the Bean's model curve. The difference in the pinning between them is clear, with relatively better pinning properties for the $SmFeAsO_{0.85}F_{0.15}$, supporting the role of the "spacer" layers in the pinning behaviour.

Regarding the 11 system, data shown in Fig. 2 provides a clear indication that the 1111 systems with "spacer" layers such as $NdFeAsO_{0.86}F_{0.14}$ and $SmFeAsO_{0.85}F_{0.15}$, are characterized by a higher pinning force and larger critical current values compared to the $FeSe_{0.88}$ containing only the "active" FeSe layers. The result is important, because the higher pinning behaviour observed in the 1111 ironpnictides contradicts the common understanding that a material with a higher critical temperature and a larger anisotropy associated to a larger thermal activation should have a lower flux pinning. As a consequence, the stronger

Table 1: Filling factors of the samples calculated with respect to volume of the pick-up coil.

	$NdFeAsO_{1-0.14}F_{0.14}$	$SmFeAsO_{0.85}F_{0.15}$	$FeSe_{0.88}$	$FeSe_{0.5}Te_{0.5}$	$FeSe_{0.25}Te_{0.75}$
FF	0.015	0.0025	0.185	0.0029	0.339

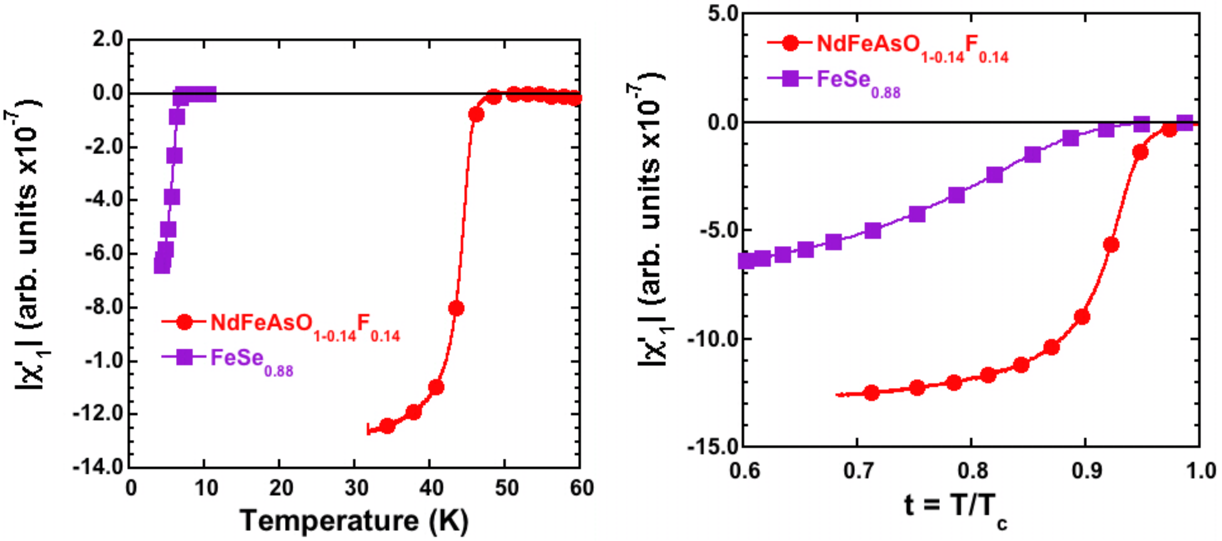

Figure 1: a) Magnitude of the first harmonic ac- susceptibility $|\chi_1|$ measured at 1070 Hz without dc field (H_{dc} = 0 T) vs. temperature (left); b) the ac- susceptibility $|\chi_1|$ as a function of the reduced temperature t=T/T_c (right).

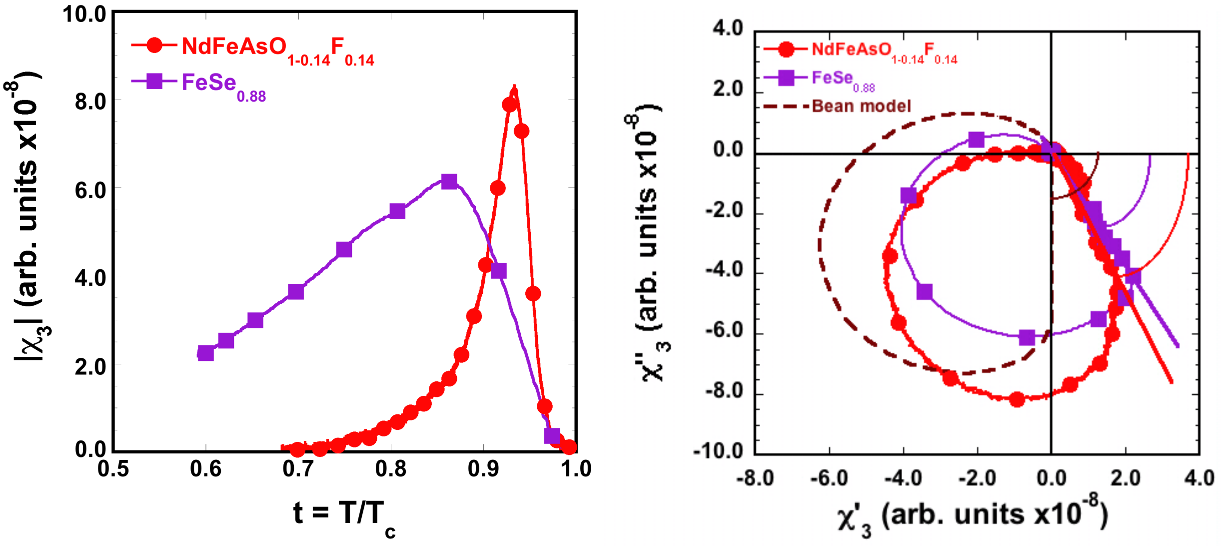

Figure 2: a) Comparison between $|\chi_3|$ (left) and; b) Cole-Cole plots (right) at 1070 Hz with no dc-magnetic field (H_{dc} = 0 T) for both $NdFeAsO_{0.86}F_{0.14}$ and $FeSe_{0.88}$.

flux pinning observed in the 1111 systems with respect to the 11 has to be associated to their structural topology and to a different atomic disorder in the FeX "active" layers. To clarify the issue, we have investigated the Te substituted FeSe 11 system, i.e., the $FeSe_{0.5}Te_{0.5}$ and the $FeSe_{0.25}Te_{0.75}$, two compounds without the 'space layer' but with a different atomic disorder in the active layer due to Te substitution. Similarly to random alloys, these two ternary systems show also a nanoscale phase separation characterized by different Fe-Se and Fe-Te bond lengths, [28, 58].

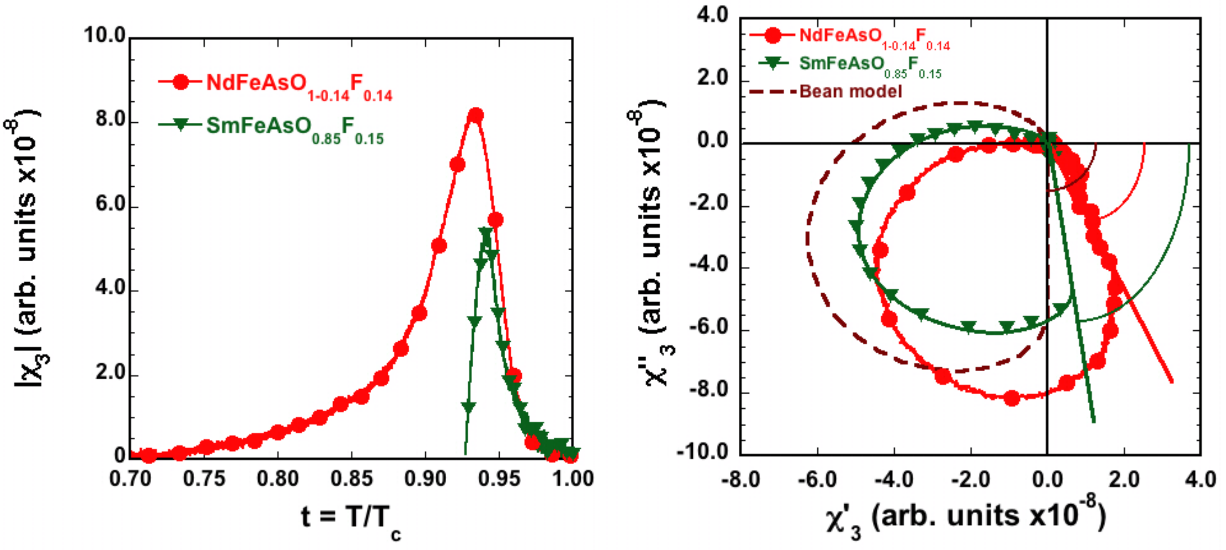

Figure 3: a) Comparison between $|\chi_3|$ (left) and; b) Cole-Cole plots (right) at 1070 Hz with no magnetic field (H_{dc} = 0 T) for NdFeAsO$_{0.86}$F$_{0.14}$ and SmFeAsO$_{1-0.14}$F$_{0.14}$.

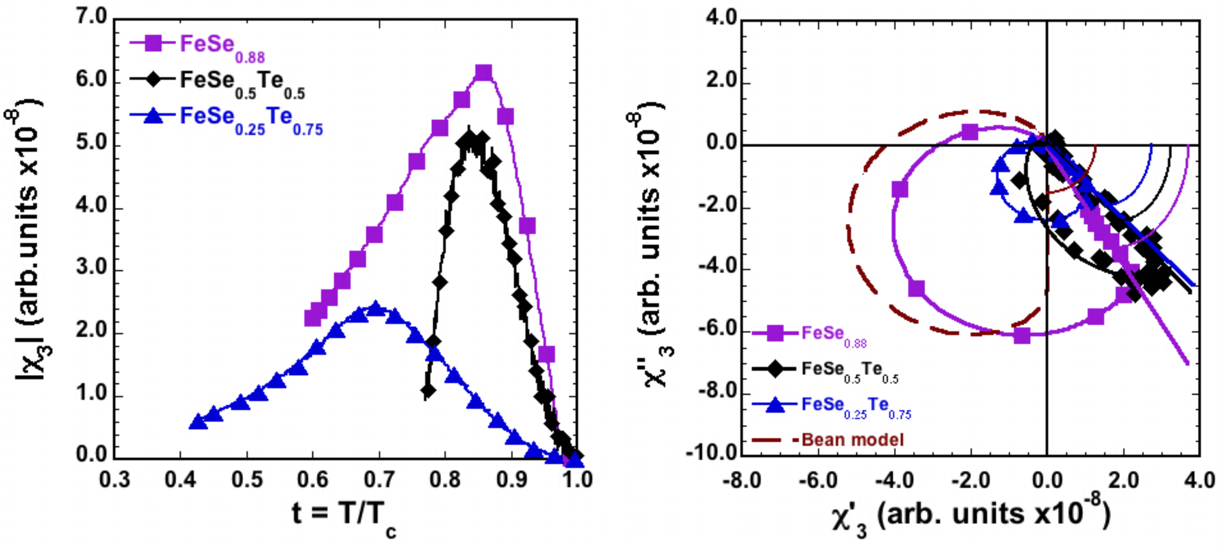

Figure 4: a) Comparison of $|\chi_3|$ (left) and; b) Cole-Cole plots (right) for FeSe$_{0.88}$, FeSe$_{0.5}$Te$_{0.5}$ and FeSe$_{0.25}$Te$_{0.75}$ at 1070 Hz and H_{dc} =0 T.

In Fig. 4 we compare the third harmonic of the AC-susceptibility and the Cole-Cole plot for FeSe$_{0.88}$, FeSe$_{0.5}$Te$_{0.5}$ and FeSe$_{0.25}$Te$_{0.75}$ samples with the Bean's model. Differences are evident, with disorder playing an important role in flux-pinning properties. Indeed, pinning forces are lower in the ternary 11 system characterized by a random disorder. Moreover, the difference in the pinning force between binary and ternary systems is large and, therefore, both the structural topology and the disorder in the active layer contribute to the different transport properties of these materials. We associate the greater flux pinning of the 1111 system to a misfit strain that induces a controlled disorder in the active FeX layer via large interlayer atomic correlations [59–61] present in this family. The scenario is different in the 11 system in which the random disorder in the FeX layer is at the origin of the weakening of the pinning force.

Fig. 5 shows modulus of third harmonic, measured using DC-magnetic field of 1 T, as a function of temperature for NdFeAsO$_{0.86}$F$_{0.14}$ [T_c(1 T) = 46.1 K] and FeSe$_{0.25}$Te$_{0.75}$ [T_c(1 T) = 9.47 K]. These data provide further support to the stronger pinning in the 1111 family with respect to the samples of the 11 family, that should be related to the presence of the spacer layer and controlled disorder in the active layer. An additional evidence of higher flux-pinning in the 1111 system is provided by the critical current measurements with high DC magnetic field on single crystal samples of $SmFeAsO_{0.7}F_{0.25}$ [62] and $FeTe_{0.61}Se_{0.39}$ [63].

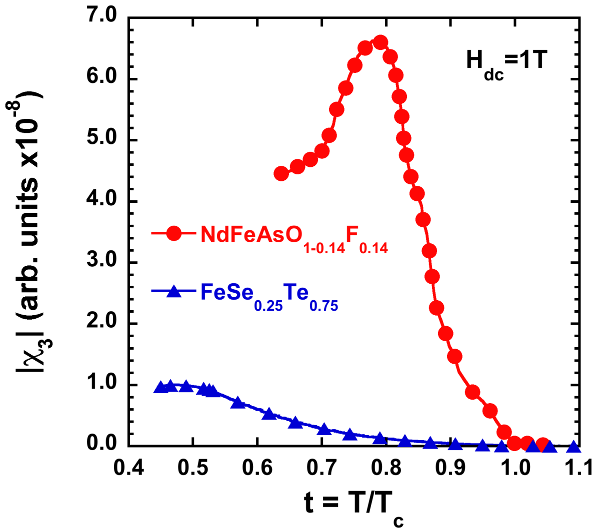

Figure 5: Comparison of $|\chi_3|$ vs. t for $NdFeAsO_{0.86}F_{0.14}$ and $FeSe_{0.25}Te_{0.75}$ at 1070 Hz and H_{dc} =1 T.

Finally the Figures 3a and 4a show a further effect: the flux pinning produced by controlled-disorder due to strain/stress by 'space layer' and/or by doping can be optimized even within the same Fe-HTSC family, that is, some compounds show a better matching between FLL and flux pinning. In fact, the FLL mobility is much larger in 1111 $NdO_{0.86}F_{0.14}FeAs$ than in 1111 $SmO_{0.85}F_{0.15}FeAs$ (Fig. 3a) and much larger in $FeSe_{0.25}Te_{0.75}$ than in $FeSe_{0.88}$ (Fig. 4a).

The stronger intra-grain pinning observed in the 1111 HTSC-Fe family compared to the 11-HTSC Fe and its technological control via correlated nanoscale defects [64] will have application relevance. However keep in mind that in the fabrication of the superconducting wires and tapes the limiting factor of the critical current are mainly the grain boundaries, then as a first step need solve this technologically relevant fact [65].

4 Summary and conclusion

In summary, we have presented a careful analysis of the flux-pinning behaviour of representative Fe-based superconductors: 1111 iron-pnictides containing spacer layers such as $NdFeAsO_{0.86}F_{0.14}$ and $SmFeAsO_{1-0.15}F_{0.15}$ systems and 11 iron chalcogenides such as $FeSe_{0.88}$, $FeSe_{0.5}Te_{0.5}$, $FeSe_{0.25}Te_{0.75}$, containing only active layers stacked together. We have showed that, under the same condition of temperature and magnetic field, iron-pnictides exhibit a stronger bulk pinning force. In fact, both $NdFeAsO_{0.86}F_{0.14}$ and $SmFeAsO_{1-0.15}F_{0.15}$ are characterized by a very sharp $|\chi_3|$ peak and their Cole-Cole plots can be well described by the Bean critical state model. Moreover, the misfit strains due to REO spacer layer controls the disorder in the active layers and hence tune the flux pinning. "The results seems to indicate" as these are not direct results, rather implication of the present findings.

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