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# Microwave Characterization of 122-Pnictides by a Superconducting Niobium Cavity

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We present first results of our microwave measurements of  $Ba_{1-x}K_xFe_2As_2$  (with x = 0.34) single-crystal pnictide, using a cylindrical superconducting niobium microwave cavity, working at approximately 26.2 GHz in the TE<sub>011</sub>-mode. The measured samples, after initial magneto-optic tests of their uniformity, are cut and glued on a sapphire rod, whose temperature can be independently controlled between 5 K and 40 K. Microwave measurements are performed by a vector network analyzer in the reflection mode, i.e. the  $S_{11}$  component of the complex scattering matrix S is measured. The loaded quality factor  $Q_{Ls}$  and the shift of the resonance position are measured during a temperature sweep, followed by calculating of the unloaded quality factor  $Q_{0s}$  and the resonance frequencies  $f_{Ls}$  and  $f_{0s}$  by means of the Kajfez procedure where the subscript "s" denotes the sample. The values of the superconducting penetration depth change  $\delta \lambda_L$  and of the surface resistance  $R_s$  are calculated approximating the single crystal sample by a flat cylindrical disk of appropriate size, based on a procedure described by Maier and Slater.

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# 1. Introduction

Microwave perturbation methods play an important role in superconductor characterization (see e.g. [1] and references therein). Such methods are relatively simple to apply; on the other hand, they are sensitive enough to detect small variations in the superconducting properties.

Usually a small sample of the superconductor is placed in the *H*-field maximum in a microwave cavity. The resonant frequency change after placing a sample into a cavity allows the penetration depth to be determined, while the quality factor change with respect to an empty cavity can be directly related to the surface resistance of the superconductor.

Both the resonant frequency and the quality factor can be measured with high precision using the Kajfez method [2]. However, the relation between measured data and the sample parameters remains still the main problem in microwave perturbation methods. As compared to other methods of analysis reported so far, in the present work we suggest a slightly different approach, in which the relevant geometrical factors are determined experimentally. Such a procedure seems particularly useful for less regular samples (e.g. parallel prisms), when analytical approximations do not exist.

Summarizing, the aim of the present work is twofold. First, we describe a new version of the microwave measurement method in which we combine analytical solutions with some experimentally determined parameters. In addition, we present preliminary results for  $Ba_{1-x}K_xFe_2As_2$  single crystals, indicating that the method described here can be applied to the characterization of superconductors such as 122-pnictides.

#### 2. Samples

We have chosen  $Ba_{1-x}K_xFe_2As_2$  as a test sample. It belongs to the so called 122 family of pnictides, where superconductivity can be obtained by applying high hydrostatic pressure [3] or by chemical substitution [4].  $Ba_{1-x}K_xFe_2As_2$  is a hole-doped superconductor, with a tetragonal unit cell and a layered structure. Please note that neither  $BaFe_2As_2$  nor  $KFe_2As_2$  are superconducting under ambient pressure.

Single crystals of  $(Ba_{1-x}K_x)Fe_2As_2$ , with x = 0.34, were synthesized at Ameslab using the high-temperature FeAs flux method [5]. These crystals are 122 iron arsenides, where barium is partially substituted by potassium. Their critical temperature  $T_c$  measured by magnetic susceptibility was approximately 35 K. The uniformity of the critical current densities of all crystals was verified using magneto-optic measurements [6] which were carried out at 30 K in perpendicular magnetic fields up to 50 mT. A magneto-optic picture of sample RS 2 is shown in Fig. 1, where light areas correspond to the penetration of magnetic flux.

## 3. Microwave measurements

Smaller, more uniform samples were cut in the middle of the sample and glued by nonadecane  $(C_{19}H_{40})$  on a sapphire rod with a diameter of 1.9 mm protruding into the Nb cavity approximately 5 mm. The 26.2 GHz niobium cylindrical cavity was attached to an insert, which was evacuated below  $10^{-5}$  hPa and cooled only by the

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thermal conductivity of surrounding copper parts, being in contact with liquid He. There is no need to have a leak-tight niobium resonator because there is no danger of liquid helium or He gas penetration into the cavity during the measurement. A picture of the niobium cavity is shown in Fig. 2.



Fig. 1. Magneto-optic images for a big sample RS 2.

A pressure stabilization device was added to our He recovery system, to prevent pressure drops caused by periodic evacuation of the gas-holder close to the He liquefier. During the measurements, the sample temperature as well as the niobium cavity temperature were monitored.

Microwave measurements were carried out using a vector network analyzer (VNA), model N5222A (Keysight Instruments). A microwave power of 0 dBm was applied, the sweep frequency width was 2 MHz around an appropriate center frequency between 26.1 and 26.5 GHz. The intermediate frequency of the sweep was set to 2 kHz. For microwave measurements carried out vs. temperature the shape of the reflection coefficient  $S_{11}$  was recorded. Since we have used a vector network analyzer, measuring the microwave amplitude and its phase, a well known calculation procedure described by Kajfez can be applied, thus allowing to calculate the loaded quality factor  $Q_{Ls}$ and the unloaded quality factor  $Q_{0s}$ , together with their



Fig. 2. Essential parts of the superconducting cavity: (a) niobium cylinder embedded into a threaded copper tube, (b) niobium cavity fully covered by a gold-plated copper housing, with 2.92 mm microwave connector and guiding post for the sample holder (white).



Fig. 3. Temperature dependence of the  $Q_0$  factor of the empty cavity with 1.9 mm diameter sapphire.

corresponding frequencies  $f_{Ls}$  and  $f_{0s}$ , based on approximately 200 points of the Smith circle. The subscript "s" denotes the cavity with sample mounted on the sapphire rod inside.

The temperature dependence of the  $Q_{0e}$  factor of our microwave cavity is shown in Fig. 3. The subscript "e" denotes the empty cavity without sample but with sapphire rod inside. The increase of the cavity temperature is caused by operating the sample heater and this effect has to be subtracted later from the sample data. At 4.9 K we have obtained a  $Q_{0e}$  factor of  $1.5 \times 10^7$ , which is typical for niobium cavities [7].

# 4. Calculations of $R_s$ and $\delta \lambda_L$

Knowing  $Q_{0s}(T)$  and  $f_{0s}(T)$ , it is possible to calculate the surface resistance and penetration depth of a superconducting sample, using simple formulae derived by cavity perturbation theory by Maier and Slater [8], who obtained analytical solutions for a conducting sample having the shape of a sphere, needle, or an ellipsoidal disk placed in a cylindrical microwave cavity, taking into account the specific properties of the electric and magnetic field components of the TE<sub>011</sub> mode as well as the geometry of the samples.

Our samples, having the shape of rectangular prisms with a thickness much smaller than its lateral size, were approximated by flat ellipsoidal disks.

Considering a thin sample of effective radius a placed in the *H*-field maximum of the TE<sub>011</sub> cylindrical cavity and using Maier's and Slater's approach one can evaluate both the frequency shift and the  $Q_{0s}$  factor change as

$$\frac{\omega_{0e} - \omega_{0s}}{\omega_{0e}} = \Gamma_1 \left[ 1 - \frac{3}{2} \left( \frac{\delta}{a} \right) \right] \tag{1}$$

and

$$\left(\frac{1}{Q_{0s}} - \frac{1}{Q_{0e}}\right) = \Gamma_2 \frac{\delta}{a},\tag{2}$$

where  $\omega_{0e}$ ,  $Q_{0e}$  and  $\omega_{0s}$ ,  $Q_{0s}$  denote the resonant frequencies and  $Q_0$  factors of the empty cavity and the cavity with a sample, respectively,  $\delta = \sqrt{2/(\omega\mu_0\sigma)}$  is the skin-depth, and the dimensionless geometric factors  $\Gamma_1$ ,  $\Gamma_2$  have to be determined experimentally using a standard superconducting sample (e.g. a niobium foil) because the influence of the sapphire rod on the electric field components close to the sample cannot be calculated analytically.

The skin depth  $\delta$  is related to the surface resistance  $R_s$  by

$$\delta = 2R_s/(\omega\mu_0). \tag{3}$$

Thus, using Eq. (2)  $R_s$  can be expressed as

$$R_s = \gamma_s \left(\frac{1}{Q_{0s}} - \frac{1}{Q_{0e}}\right),\tag{4}$$

where

$$\gamma_s = \frac{\omega\mu_0 a}{2\Gamma_2} \tag{5}$$

Similar results were obtained by Sridhar and Kennedy [9].

The results of  $R_s$  and  $\delta\lambda_{\rm L}$  for our sample RS 2 are shown in Fig. 4 and Fig. 5. The expression  $\delta\lambda_{\rm L} = \lambda_{\rm L}(T) - \lambda_{\rm L}(0)$  denotes the change of the London penetration depth in a superconductor and can be calculated using Eq. (1) with  $\lambda_{\rm L} = \delta/2$  [10]. We can clearly observe a steep change of  $R_s$  and  $\delta\lambda_{\rm L}$  below the superconducting transition temperature, followed by a much slower variation vs. T at lower temperature. However, due to the unconventional nature of the superconductivity of 122 pnictides, the obtained results for both  $\delta\lambda_{\rm L}$  and  $R_s$  cannot be fitted by curves derived from the Bardeen, Cooper, and Schrieffer theory for conventional, low- $T_c$ superconductors.



Fig. 4. Temperature dependence of surface resistance of a small RS 2 sample at 26.2 GHz.



Fig. 5. Temperature dependence of the penetration depth change of a small RS 2 sample at 26.2 GHz.

## 5. Conclusions

In this work we have presented a new version of the microwave perturbation method for the characterization of superconducting materials. As compared to earlier papers on similar subject, our approach is different in some points. First, we take advantage of the Kajfez method for precise determination of the unloaded resonant frequency and the unloaded Q-factor of a cavity with and without a sample. Second, our data treatment is slightly different from that described previously, since we determine some important geometrical factors experimentally,

making it possible to characterize also samples of less regular shapes. The performance of a high  $Q_0$  niobium cavity with a  $Q_{0e}$  factor of  $1.5 \times 10^7$  at 4.9 K was clearly demonstrated. It can be applied for the measurements of surface resistance and relative penetration depth changes of small superconducting crystals above 5 K.

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