Synchrotron Radiation in the Infrared and its Application for Solid State Investigations

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The properties of synclirotroii radiation in tlie domain of tlie infrared and far-infrared are reviewed and its advantages over conventional sources are discussed. Tlie new infrared facility n Orsay (SIRLOIN), whicli is positioned on a wiggler. is described. A review of infrared optical experiinents in solid state pliysics is presented, with emphasis on measurements wliere InfraRed Synchrotron Radiation (IRSR) appears to provide major improvements over conventional spectroscopy. In particular, we discuss: I) studies of excitonic spectra in molecular crystals under pressure. II) shallow donor levels and insulating-to-metal transitions in semiconductors, III) investigations on anomalous Drude reflectivity in untwinned High-T_c superconductors, IV) gap determinations in thin superconducting siiigle crystals.

I. Introduction

Radiation emerging from bending magnets in synchrotron and storage rings is increasingly utilised as a far-infrared source. Infrared synclirotron Radiation (IRSR) facilities are operating at the NSLS VUV in Brookhaven[1] (U.S.A.) and on tlie UVSOR[2] and SPring-8[3] acilities (Japan). Further beamlines are under construction or testiiig in Daresbury (UK)[4], at tlie MAX1 ring of Lund[5] (Sweden), and again at Brookhaven. Indeed, sucli sources are by two or three orders of magnitude brighter than a black body emitter at 2000 K, at all infrared wavelengths. On tlie other hand, the total plioton flux f (number of photon/second/unit bandwidtli) is considerably greater than that eniitted by a black body for wavelengths $\lambda >$ 100 μm only[1,2]. Therefore, use of IRSR from bending magnets has been proven to be advaiitageous for measurements on small samples and/or in tlie 10000-100 μm (1-100 cm^{-1}) range. Tlie gain in brightness in this spectral region has interest pertaining at numerous fundamental fields, as for example studies of excitation spectra in crystals under pressure, or determinations of tlie supeiconducting gap in thin single crystals as described further.

An alter native source of IRSR is a wiggler, whicli may present advantages in terms of flexibility, plioton flux, and extraction optics. Such a choice has been done at the Super-ACO ring of Orsay, where tlie infrared bearnline SIRLOIN is now under final test[6]. Tlie calculated intensity of SIRLOIN, as explained in tliis paper, is significantly greater than tliat of a black body or of a bending magnet of the same ring, therefore extending tlie range of applications of synclirotron radiation. For example, tlie study of Drude anomalies in High-T_c supercoiiductors requires untwinned crystals, which drastically liinits their size, and a source with broad spectral range extending up to tlie mid-infrared.

In tlie first part of this paper we describe tlie beam line extraction system and optics, as well as tlie results of calculations of plioton flux. In tlie second part me present a few experiments tliat have been or could be performed by IRSR, emphasing its advantages with respect to conventional sources.

II. Calculation of infrared synclirotron radiation flux

II.a) Calculation of the plioton flux from an undulator in tlie "wiggler mode"

Let us consider an undulator, U, of length L and magnet period λ_u. It generates a static magnetic field

$$B(z) = B_0 \cos(2\pi z/\lambda_u) \qquad (1)$$

wliere z varies froin 0 to L along the axis of the undulator. When relativistic particles pass through U, they oscillate in the plane of tlie ring with an amplitude Δx which is comparable witli tlie transverse dimension of the beam itself. At aiiy point of tlie particle sinusoidal path, radiation is emitted tangentially to the trajectory. Tlie resulting light beam propagates as a cone coaxial with tlie particle beam.

In the "undulator mode" tlie $2N$ magnets (N periods) of U caii provide along tlie axis a very bright quasi-monochromatic plioton beam, due to tlie occurrence of a N^2 enhancement factor. The enhancement is determined by tlie interference between electromagnetic fields cmitted by the same pnrticles along its trajectory (contributions from different particles add up incoherently). In tlie plane of the ring, the first harmonic wavelength λ_1 of such an undulator may be tuned by varying either the magnetic field B_0 and/or the charged particle energy

$$\lambda_1 = 13.056\lambda_u[1 + (K^2/2)]/E^2 \qquad (2)$$

where λ_1 is given in Å, A, in cm, E is tlie particle energy in GeV, $K = 0.934\ \lambda_u B_0$ is the deflection parameter and $y = E/mc^2$.

On tlie otlier hand, whenever

$$\lambda_1 \gg \lambda$$

the coherence effects disappear, tlie photon flux is simply enhanced by a factor $2N$ and tlie undulator is used in tlie "wiggler mode".

In turn[7], when measured off-axis equation (2) becomes:

$$\lambda_1 = 13.056\lambda_u[1 + (K^2/2) + \gamma^2\vartheta^2]/E^2. \qquad (3)$$

Infrared synchrotron radiation is mainly collected off-axis and, as shown by Eq. (3), λ_1 rapidly increases with increasing 3. By introducing the parameters which hold for Super ACO[6] (y = 1565.55, K = 6.9. λ_u = 12.9 cm) into Eq. (3). one finds tliat tlie incoherence condition is fulfilled for any A $\ll$ 58.7 μm if ϑ = 30 mrad. Tliis rcsult shows that in thc infrared tlie radiation is incoherent, while coherence effects could in principle be observed in the far infrared region.

However, tlie observation of colierence is unlikely even at long wavelengths for the following reasons : I) radiation is collected under a. wide acceptance angle, thus causing a. superposition of spectra witli different λ_1, according to (3); II) e.m. fields einitted at points separated by A, $2\lambda_u$, $3\lambda_u$, ... will be intercepted by the collecting mirrors under different angles, as those are placed at a finite distance from tlie undulator (approximatly 1.5 m from tlie end of tlie undulator). This also considerably reduces spatial coherence.

From the above discussion we conclude that the incolicrent "wiggler mode" may be assumed as valid in tlie present case. Iii order to calculate tlie total photon flux as a function of tlie radiation wavelength A, we decompose the undulator length L into 3096 intervals corresponding to an element Δz of 1 mm. Any such interval corresponds to an element $\Delta 1$ on tlie beam path. In turn one lias $\Delta 1 = r\Delta\theta$, where r is an instantaneous radius of curvature. In such a way, tlie sinusoidal positron trajectory is decomposed into elementary circular paths of variable radius r and variable aperture AO. Tlie photon flux emitted by positrons from any trajectory element $\Delta 1$ at position z can then be calculated by tlic standard formulas for a bending magnet which produces a magnetic field $B(z)$ as given iii Eq. (1). The total photon flux will then be obtained by a suitable integration over z.

II.b) Calculation of plioton flux for SIRLOIN

The number of photon $\Delta\Phi_B$(A) emitted per milliradian angular path $\Delta\theta$ is given by :

$$\frac{\Delta\Phi_B(\lambda)}{\Delta\theta} = 2.457 \times 10^{13} E\ I\ y \int_y^\infty k_{2/3}(y')dy'. \qquad (4)$$

Here E is tlie beam energy in GeV, I is tlie beam current in Ampere, $y = \lambda_c/\lambda$ (where A, is tlie critical frequency of synchrotron radiation) and $K_\nu(\lambda)$ is a modified, second-order Bessel function. Eq. (2) is then numerically evaluated under tlie assumption that the transverse displacement Δx of the point source in tlie undulator, being of the same order of magnitude as tlie beam width, may be neglected. Tlie results are shown in Fig. 1 wliere the infrared photon flux Φ calculated for tlie SU3 undulator of Super ACO. is compared with tlie one calculated for a bending magnet of the same ring. In the latter case, we assumed that radiation is extracted from a A0 = 50 mrad section of the orbit and is integrated in the Vertical plane. The flux Φ emitted

hy tlie uiidulator turns out to be greater by more than one order of magnitude at all wavelengths . Tlie flux einitted by a 10 mm^2 black body at 2000 K under a solid angle of $50 \times 50 mrad^2$, wliicli can be considered as usual in conventional devices, is also sliown for comparison. It should also be considered that globar sources in standard spectrophotometers are kept at temperatures ranging from 1300 to 1500 K.

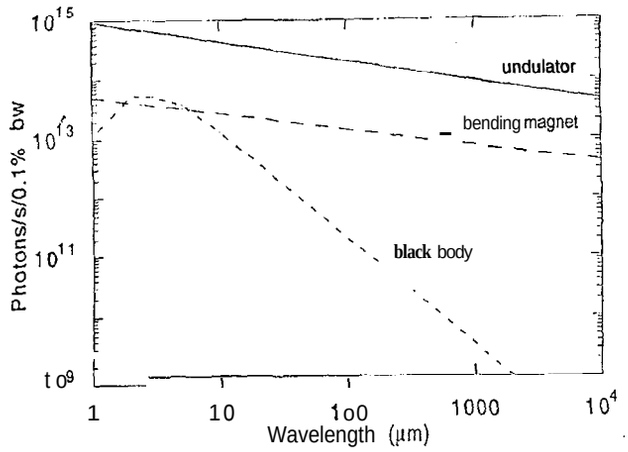

Figure 1: Infríred photon flux, as calculated for a 10 mm^2 black body at 2000 K under a solid angle of 50x50 $mrad^2$ (dotted lirie), for IRSR produced by a bending magnet with AO = 50 mrads (dashed line), and for IRSR emitted by the undulator SU3 with K = 6.9 (solid line) and a current of 200 mA injecttd.

The geometrical distribution of tlie radiation intensity, as effectively available at the extraction mirrors of SIRLOIN after taking into account the geometrical constraint of the undulator, is pictorially represented for a wavelength of 100 μm in Fig. 2 therein, each darker tone of grey corresponds to an intensity loss of 12,5% coinpared to its maximum value (white). Tlie dotted lines represent tlie plane mirrors witli a standard separation of 25 inin. They allow to evaluate the fraction of photon flux which is intercepted and transmitted to the line. Sucl fraction turns out to be about 80% in Fig. 2 and reiluces to about 30% for a wavelength of 1 μm. It can ɔe noted in Fig. 2 that the infrared intensity presents a minimum at tlie centre of the ring, which makes the geometry here employed particularly suitable for the extraction of IRSR.

In fig. **3,** the calculated plioton flux being intercepted by the plane mirrors of SIRLOIN is plotted as a function of the radiation wavelength for different values of A. For comparison, tlie flux emitted[6] by a black body at 2000 K is also sliown. Finally, we remark tliat even hetter performances would be obtained if tlie undulator were expressly designed for IRSR.

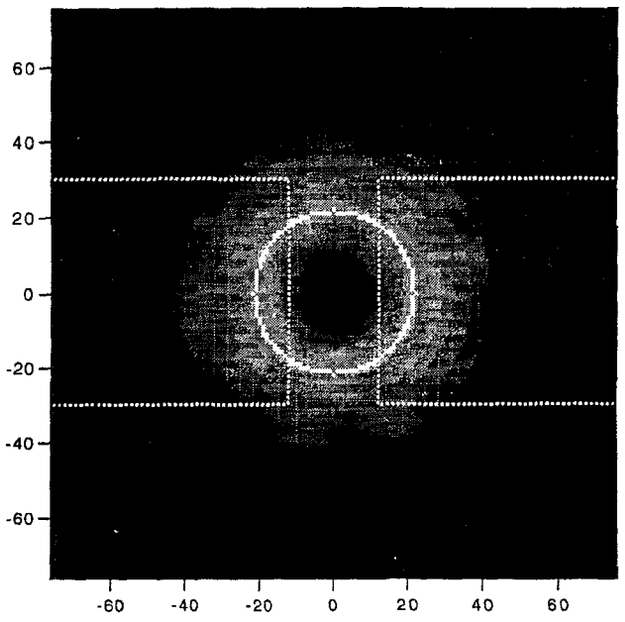

Figure 2: Distribution of the plioton flux on mirrors M_1 and M_1' (repcesented by the dotted lines with scales in millimeters) at a wavelengtli of 100 pm and for a typical gap A = 25 *mm.* Each tone of gray from white to black corresponds to a 12,5 % loss with respect to the maximum intensity.

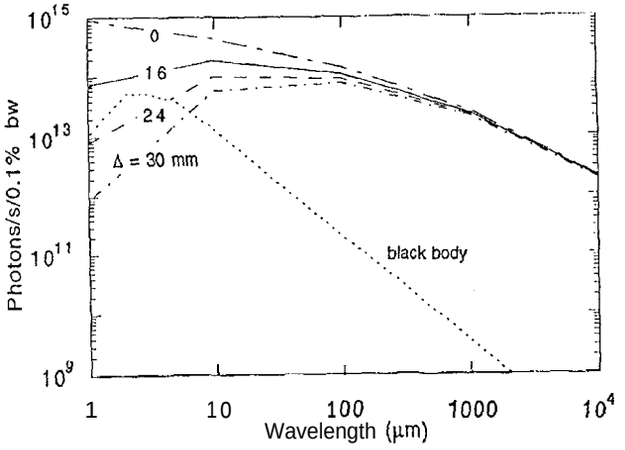

Figure 3: Intensity of tlie photon flux intercepted by tlie mirrors at tlie exit of the undulator SU3 of Super Aco, as calculated for various mirror separations A and compareci ta tlie emission of a black body (10 mm^2 black body at 2000 K collected under a solid angle of 50×50 $mrad^2$).

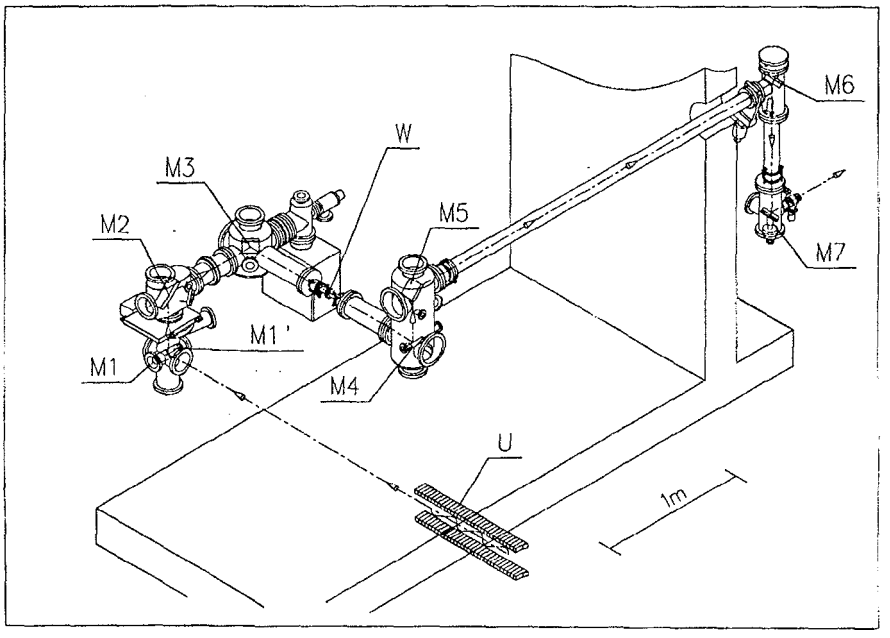

Figure 4: Scliematic view of SIRLOIN, tlie IRSR liiie whicli is presently beiiig assembled on tlie Super Aco storage ring of LURE. Wide-angle, low energy radiation produced by positrons in tlie undulator U is reflected vertically by plane mirrors M_1 and M_1' wliile tlie positroiis and tlie hard part of tlie spectrum passes in between. Tlie beam is tlien focussed by tlie cylindrical mirrors M_2 and M_3 on tlie diarnond window W, and matclied to tlie entrance optics of tlie interforemeter by mirrors M_4 to M_7. The diamond window W, which lias an aperture of 1 cm, separates tlie ultrahigh-vacuum section of tlie line from tlie low vacuum of tlie iiiterferometer.

III. Tlie SIRLOIN spectroscopic system

Tlie SIRLOIN beamline is schematically shown in Fig. 4. Its extracting mirror is assembled on oiie of tlie eiglit straiglit sections of tlie Super ACO storage ring, whicli contains tlie variable-gap undulator SU3 (U in Fig. 4). Tliis latter lias a length L = 3096 mm, a maximum $B_0 = 0.57$ T, aiid a magnet period $\lambda_0 = 12.9$ cm. The undulator is currently used in the wiggler mode, witli B_0 corresponding to $K = 6.9$ for tlie 800 MeV positron energy[8].

In the following description of tlie optical systein, tlie source point is considered as being tlie centre of tlie undulator. I-Iowever it is clear that tlie positrons, whicli emit photons througli all tlieir 3 meter trajectory in tlie undulator, cannot be considered as a point source. This remark holds for any synchrotron line for whicli a large collecting angle is involved.

At about 1 m from the output of the undulator two gold-coated, copper plane mirrors (M_1, M_1') are placed at 4° with respect to the z axis. Tlie mirrors are water-cooled and are kept at a certain separation A in order to let positrons, X, and UV radiation pass through. In such a way tlie IR beainline does not interfere witli the existing oiies, aiid in standard conditions the beam lifetiine is not appreciably affected. Moreover, it is desirable to set tlie mirror separation A at its maximum value during tlie injection of positrons into tlie ring, when tlie beain is scarcely collimated. Tlie mirror displacements are performed by a remote-controlled device. It is designed to move eitlier mirror liorizontally while keeping both parallel within a few milliradians, and while keeping tlie pressure in tlie ring at 5×10^{-11} torr. Such requirements have been fulfilled during tlie tests[6].

Infrared aiid visible radiation is then focused onto tlie type 2A natural cliamond window W by cylindrical mirrors M2 and M3, whicli have tlieir axes in the horizontal and vertical planes, respectively. Tlie window is 200 microns thick, has a usable diameter of 10 mm and is wedged. Tlie window separates tlie ultraliigli vacuum zone froin tlie remainder of tlie line, which is equipped witli plane (M_5 aiid M_6) and toroidal (M_4, M_7) mirrors. Tliis section is Bept at $\approx 10^{-3}$ torr in order to strongly reduce tlie infrared absorption due to water and CO_2. The line ends in correspondence with the

"emission" port of a rapid scanning Bomem DA8 3X interferometer. The centre of tlie undulator (considered as tlie source point) is focalized on tlie last toroidal mirror M_7 wliile tlie M_1 extractioii inirrors (considered as collimators) are focused onto tlie 10 mm entrance port of the interferometer. From there on, synclirotron radiatioii follows exactly tlie same optical patli as tlie interna1 sources of tlie interferometer. This will allow a direct comparison of the intensity aiid briglitness of IRSR witli the well known equivalent quantities which can be measured from a black body.

IV. Some applications of IRSR to solid state pliysics

IVa. Studies on inolecular aiid ionic crystals at high pressures

Infrared synchrotron radiatioii lias been successfully used fo: performing high-pressure measurements in a diamond anvil cell (DAC) in Okasaki[9] and in Brookhaven[10]. Such experiineiits are difficult witli conventional light sources, because of the small size of tlie windows.

Nanba and Watanabe[11] measured tlie cliange in tlie optical pionon spectrum of a KI crystal in the 10-250 cm^{-1} range for the transverse optical phoiion (TO). Tlie variation in frequency ε as pressure p increases (Fig. 5) allovrs to calculate tlie Gruneisen parameter

$$\gamma = (d\varepsilon/dp)_T/k_T\varepsilon, \qquad (5)$$

wliere k_T is the isothermal compressibility. 7 is critically dependent on the shape of tlie repulsive part of tlie inter atomic potential. Its spectroscopic measurement can be usefully compared to thermodynainic or macroscopic determinations, and provides detailed information on the potential at the atomic scale. In tlie KI system, a phase transition at 1.8 GPa from tlie rock salt structure to tlie caesium chloride has been observed by Nanba et al. using IRSR at tlie UVSOR[11].

Phase transitions are also found in molecular crystals at liigli pressures[12]. An example is given by solid CH_4, where at least five phases have beeii observed, for which th: orientational and/or the crystal structure differ. The corresponding changes in tlie Gruneisen parameter can be monitored in diamond anvil cell by IRSR absorption. One should notice that determinations of the Gruneisen parameter are also crucial in the assignment of the observed propagating modes in inolecular crystals. In general, translational phoiions present γ values considerably higher than vibrational modes[13].

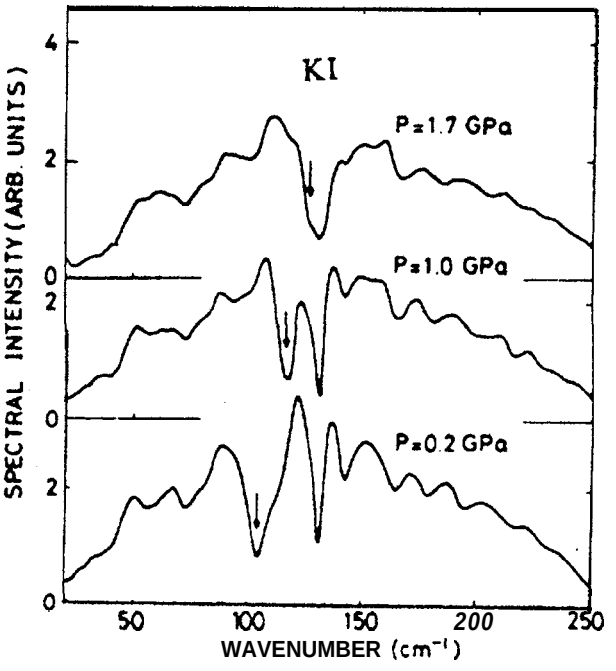

Figure 5: Transmission spectra of KI evaporated film of 2 μm thickness measured at room temperature under liigli-pressure using a diamond anvil cell. Tlie pressures monitored by tlie energy shifts of the R1 fluorescence line of ruby are sliown on spectra. Sharp dip structure due to tlie absorption of tlie TO plionon mode of KI crystal are shown by downward arrows. Structure at 130 cm^{-1} is due to tlie absorptioii of tlie crystalline quartz filter of tlie detector (from reference 9).

More recently Hanfland et al.[10] also used synchrotron radiation for measurements of infrared spectra of samples at ultraliigh pressures. Tlie measurements of vibrational transitions of solid hydrogen up to 180 megabar pressure revealed the meclianism for weakening tlie intrainolecular interactions aiid strengtliening tlie intermolecular interactions.

Altliougli some of the above measurements could be accessed by Raman spectroscopy, tlieir interpretation in IR spectra is more straightforward as they are not complicated by interference from visible absorption and fluorescence of the stressed diamond anvils. Moreover, in crystals where a centre of inversion is present, Raman active phonons are not infrared active and vice versa. As far as molecular vibrations are concerned, they are active in botli kinds of spectra, but infrared absorption often provides better signal/noise ratios[10].

IVb. Optical study of insulator-metal transition in semiconductors

Doped semiconductors represent an ideal system of hydrogenic atoins with one electron per site in wliich tlie transition from an insulating to a metallic state can be studied by varying tlie conceiitration of impurities. The cliange in the behaviour of electrons, which pass from localized to itinerant states, can be suitably monitored by optical spectroscopy in tlie far infrared. An example of a host systein is Si, which presents characteristics of high purity. When iiicreasing the impurity conceiitration, the absorption spectra change from a series of sharp lines into a unique broad band typical of metallic state. In order to refine tlie tlieoretical models one should study sainples with concentrations as close as possible to the metallic concentration. Tliis implies tlie needs of a more intense far infrared source than the conventional ones used for tlie previous studies and briglit enough to overcoine the limitations due to tlie small sample dimensions and reduced transmittance[14].

IVc. Investigation of tlie anomalous Drude behavior of HTS

It is well known[15,16] that the complex dielectric function $\epsilon(\omega)$ of High. Tc superconductors (IITS) in tlie normal-state ($T \geq T_c$ where T_c is the superconducting critica1 temperature) does not follow the ordinary Drude law

$$\bar{\epsilon}(\omega) = \epsilon_1 + i\epsilon_2 = \epsilon_\infty - \frac{\omega_p^2}{\omega^2 + i\omega\Gamma_D} \qquad (6)$$

Here ϵ_∞ includes all transitions at higher energies, while the Drude term includes plasma frequency ω_p and width Γ_D.

Infrared studies on single crystals belonging to different famillies of HTCS[17]: $La_{2-x}Sr_xCuO_4$, Nd_{2-x} Ce, CuO_{4-y}, Pb_2 Sr_2 (Eu,Dy) Cu_3 O_8, $Bi_2Sr_2Ca_nCu_{n+1}O_{2n+4}$, $YBa_2Cu_sO_{7-y}$ have sliown that Eq. (6) does not fully describe their spectra in the normal phase, as an extra-Drude absorption is always observed in the mid-infrared. Two different phenomenological approaches have been used to describe this effect. In the former approach, tlie optical conductivity $\sigma(\omega) = (\omega/4\pi)\epsilon_2$ at $\omega < 1$ eV is modeled by a "pseudo-Drude" term wliere both tlie effective mass of tlie carriers and their scattering rate depend on w. Among the possible interpretations, incoherent scattering in a Marginal Fermi Liquid[18], lias been proposed. According to an alternative approach, the dielectric function $\epsilon(\omega \leq \omega_{gap})$ is decomposed into an "ordinary Drude" term (where all parameters are independent of frequency) plus an extra-Drude term modeled by one or more Lorentz oscillators centered in tlie mid-infrared (MIR band):

$$\bar{\epsilon}(\omega) = \epsilon_\infty - \frac{\omega_p^2}{\omega^2 + i\omega\Gamma_D} + \frac{S_{\mathrm{MIR}}^2}{(\omega_{\mathrm{MIR}}^2 - \omega^2) - i\omega\Gamma_{\mathrm{MIR}}}. \qquad (7)$$

Tlie MIR independent contribution in Eq.(7) could be due to interband transitions or to polaronic effects. In any case, tlie understanding of sucli extra-Drude absorption inay provide important liints on tlie comprehension of tlie ineclianism of superconductivity in BTCS. As a preliminary indication, recent infrared measurements have pointed out a connection between this anomaly and the critical temperature of High-T_c cuprates[17,19].

A suitable material for such studies is YBa_2 Cu, O_{7-y} (YBCO), where single crystals witli high reflectivities, liigh critica1 temperatures, and intense Drude terms are commonly obtained. Nevertheless, in sucli sainples tlie main part of the surface presents twiiined crystals, wliich in turn cause ambiguities in the interpretation of physical information. Untwinned crystals are presently available only iii very small sizes (typically 100 x 100 μm^2). Therefore tlie conclusions of recent studies by microscopic infrared spectroscopy are coiitroversial. For instance, Rotter et al.[20] find an extra-Drude absorption peaked at 0.25 eV cm^{-1} and assign it to the Cu-O chains of YBCO, while Koch et al.[21] do not find any extra-Drude contribution from the chains and only observe a MIR band from Cu-O planes centred at 0.58 eV.

This type of studies is again a clear example of a situation where increased briglitness would iinprove tlie reliability of experimental data. Another example is given by tlie investigation of tlie optical properties of single crystals along the c axis of the tetragonal structure. In this case, the difficulty is represented by the small thickness of most single crystals obtained up to now. Only $La_{2-x}Sr_xCuO_{4-y}$ single crystals, thick enough for accurate reflectivity measurements by conventional sources have been recently produced[22], allowing the discovery of a superconducting phase along the c direction.

IVd. Determination of the superconducting gap in HTCS

According to the BCS theory in the weak-coupling approximaticn ($\lambda = N(E_f)U < 1$, where $N(E_f)$ is the density of electron states at the Fermi energy, and U is the averagt: electron phonon interaction), one has for the superconducting gap 2A the expression

$$2\Delta/kT_c = x \approx 3.5 \qquad (8)$$

where T_c is tlie critical temperature. Larger values of x are predicted when strong- coupling corrections are introduced into the BCS theory. An unambiguous determination of t ıe gap in High-T_c superconductors would then represent an important test for the theory, as it would inform whether: i) HTCS can be explained in the context of the BCS theory, ii) if yes, how strong the electron-phonon coupling may be. Attempts of measuring A havt been made from the beginnings both by infrared reflectivity and tunnel experiments, leading to contradictory results: indeed, x values scattering from 3 to 8 and even more, were reported in the literature.

In the far-infrared, the opening of a gap at $T < T_c$ corresponds to the appearance of an edge in the reflectivity $R(\omega)$ a: a photon energy close to 2A. In real systems, two conditions have to be fulfilled in order to perform meaningful measurements : I) the sample must have $R(\omega) = 1$ for $w \to 0$ in the normal phase, ii) the sample must be in an intermediate situation between the so-called 'clean limit" and the "dirty limit"[16]. In such case one will observe $R(\omega) = 1$ for any $w < 2A$, while for $w > 2\Delta$, $R(\omega)$ will follow a Drude behaviour.

Transmissiɔn measurements, even if are also subjected to constraints, are more straightforward. The quality of sample surface is not quite as critical. Moreover, the edge would correspond here to the transmittance changing from a zero value to a finite value, and does not imply the definition of an absolute reference as in the case of reflectivity measurements.

Thin films cannot be used for far-IR transmission measurements as phonon absorption from the substrate would mask ar y change in the transmittance related to the opening of a gap. On the other hand, free standing crystals have low transmittance (from 0.1 to 0.2) and limited size. High brilliance is then requested. Pioneering measurements of $Bi_2Sr_2CaCu_2O_x$ in the region of the ga) by means of IRSR sources have been reported[23], even if no gap opening has been evidenced for $T < T_c$ (Fig. 6).

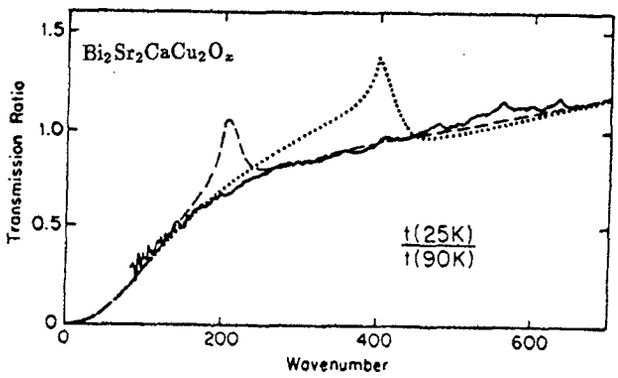

Figure 6: Ratio of the transmissions of $Bi_2Sr_2CaCu_2O_x$ at 25 and 90 K. The solid line represents the measured transmissions. Tlie dashed and dotted lines were obtained from calculations. Tlie Drude relaxation rate used was 50 cm^{-1} at 25 K. The BCS gap was assumed to be $2A = 3,5\ K_BT_c = 200\ cm^{-1}$. The calculation predicts a transmission peak even for $1/\tau < 2A$, no peak is seen in the measurement (from reference 23).

V. Concluding remarks

The extraction system of SIRLOIN is new in its concept as for the first time the collecting mirrors are placed directly on the trajectory of the positrons themselves and as it allows two regions of the spectrum (the soft X-ray and the infrared) to be exploited in parallel. In this study two important questions concerning such a device have been answered. Firstly, how small can the gap between the two mirrors be before the positrons beam is significantly affected. Secondly, according to calculations what fraction of radiation can be collected in the infrared and far-infrared range.

It should be remembered that all the calculations are based on the assumption that coherence effects are negligible due either to the very large divergence and to the length of the source. We are currently developping a program that would allow to include such effects. Another desirable improvement would be the calculation of the degree of polarisation of the light.

Moreover, measurements of the infrared intensity as compared to the radiation emitted by a black body source is being completed. This will allow in turn a comparison to the calculated intensity. This comparison extended for different values of the undulator parameter K and for different gap values should allow a deeper understanding of the undulator emission for longer wavelengths. After the optimization of the optical system the scientific program will then start with

emphasis on experiment for which synchrotron radiation brightness aiid intensity may provide information tliat could not be accessed with conventional sources.

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