Construction of the Magnetic Phase Diagram in Small-volume HTS by an OFC Magnetometer

S.G. Gevorgyan, T. Kiss, M. Inoue, T. Ohyama, M. Takeo, T. Matsushita, and K. Funaki

***Abstract*—We applied the OFC magnetometer for studies of vortex dynamics in small-volume HTS. The substitution of the solenoid coil by a flat one in a tunnel diode oscillator made the coil's filling factor maximal for flat specimens enabling to reach 1-3Å absolute and 10^{-6} relative changes of the penetration depth, λ, of RF magnetic field into HTS films. Our technique operates in high magnetic fields and measures linear changes of λ in 1mm range with high resolution. It is also an excellent Q-meter for detection of changes $\sim 10^{-9}$W of the absorption in HTS samples. Such abilities allow it to be applied in many fields of science and technology. In particular, detection of the magnetic field-dependent changes of the frequency and amplitude of a 23MHz oscillator enabled lines on the magnetic phase diagram for Y-Ba-Cu-O film to be constructed. It seems to have many similarities as compared to the diagrams defined for bulk HTS by the other methods. However, how close is the relation in fact, may be revealed after additional tests on the same quality specimens.**

***Index Terms*— flux dynamics, magnetic phase diagram, small-volume HTS, open-flat coil (OFC) magnetometer.**

I. Introduction

FOR various applications of small-volume plate-like HTS the construction of the magnetic phase diagram over wide ranges of temperature and magnetic field, preferably during a simple experiment, is a task of primary importance at present. In this context the penetration depth, λ, is a useful testing parameter, which is sensitive to changes induced by the external fields and besides, it characterizes the superconducting state from many sides. In the case of magnetic field-dependent studies on λ, the experiments are performed by use of the resonant methods, where sample is placed in a resonator or near it, or forms a part of a testing resonator. It is so, because the changes in λ vs. the field are small for many studies as compared to its zero-field value, and thus, high resolution is required [1]. Comparison among the methods for λ tests permits to conclude that the "LC resonator" method, used in a frequency range of MHz, has advantages [2]. Due to low frequency it enables to avoid the quasiparticle excitation by a testing field, which is important at studies near T_c. But, it is suitable for tests in bulk samples only. Massive LTS crystals used in this technique allow to increase the filling factor and reach an excellent absolute resolution ~0.2Å (with a good relative resolution $\Delta\lambda/\lambda \sim 2\cdot 10^{-4}$) [3]. This method, however, gives the resolution at most 10–20Å [1] when it is applied to the plate-like HTS, because of low filling factor of solenoid coils arising from the small sample volume. To overcome the problem one can approximate the coil size to the sample dimension [4]. But this can't be final decision for a task, especially for the films with a thick substrate. We developed an improved "LC resonator" technique (named in [2] as "OFC magnetometer" for λ tests in such small-volume flat HTS. It has $\Delta\lambda \sim 1$Å ($\Delta\lambda/\lambda \sim 10^{-6}$) resolution, attainable with SQUIDs, enough to solve the task. Besides, it is simple, can operate in wide ranges of temperature and magnetic field and measure in wide dynamic ranges of the testing parameters. Its better resolution, compared to others, becomes evident especially at tests near T_c. All the above factors allow to apply this technique in many fields of science, instrumentation and advanced technology, to create new types of testing and controlling devices and for basic study. In particular, we are using it at present to study the peculiarities of the vortex motion in plate-like HTS specimens near T_c. Here we report on the possibility to construct some lines on the magnetic phase diagram for Y-Ba-Cu-O film by this method, illustrating the usefulness and usability of the improved "LC resonator" technique.

II. Experimental Method

So, we applied the improved technique for high-resolution detection of the magnetic transition curves (the frequency and amplitude vs. the field at the S/N transition) in Y-Ba-Cu-O films. It differs from the traditional technique [3], [5] by a replacement of the solenoid testing-coil by the flat (open-faced) one driven by a stable-frequency tunnel diode (TD) oscillator [2], [6]-[7]. Fig.1 shows the schematics of our technique. The changes enabled to make the coil's filling factor about a unit for flat specimens, resulting in an increase of the resolution of this method by 4 orders of magnitude. For comparison, the typical values of a filling factor for solenoid coils are 10^{-3}–10^{-4} for film samples. In the new technique the measuring effects are determined by a distortion of the coil testing field's configuration near its face and also by the absorption of the field's energy by a sample (due to external influences).

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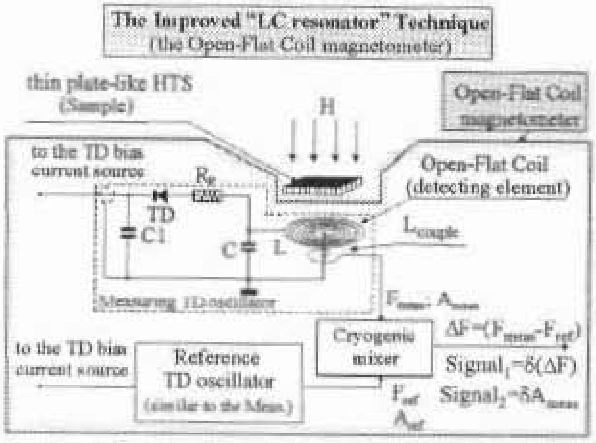

Fig.1. Schematics of the OFC magnetometer with a flat HTS specimen

These result in the changes of the oscillator frequency or/and amplitude. Compared with a traditional method, in this technique the field is densely distributed near coil. Besides, due to flat geometry (providing maximal filling factor) the flat sample at its S/N transition distorts it much strongly during the penetration of the flux of a testing field, generated by a coil, into the sample. And also, the insignificant changes in testing field's power absorption by a sample result in far stronger changes of the oscillator amplitude in this technique with a flat coil. It is also due to dense field distribution near coil.

In order to determine vortex-related parameters in HTS specimen pressed to the face of the flat coil, it is necessary to measure both the shifts in frequency and the changes in Q-factor of the resonant circuit. These data, extracted from the tests of the frequency and amplitude, will give complete information on the impedance of the sample. But, for correct determination of the physical characteristics of samples by this technique there is a need to calibrate properly both the shifts in oscillator resonant frequency (inductance) and the changes in its amplitude (Q-factor, losses). In this sense the problem here is far more complicated as compared to the solenoid coil-based technique, because even for the simplest case of a thin plate-like sample the calibration data are strongly dependent on the sample's position and size.

Due to page limitation we present here the frequency calibration data only enough, for discussion the results (more on the calibration see [8]). The measured change in oscillator frequency, δF, can be connected with the length, λ_S, of magnetic shielding by a sample of the testing field generated by a coil: $\lambda_S = -G \cdot \delta F$. In general, the shielding length λ_S, we deal at the tests, is not the same as the London penetration depth, λ_L, in Meissner state or the pinning depth, λ_{pin}, in mixed state. But, the lengths λ_S and λ_L may, apparently, be identified. On the other hand, if the sample is in mixed state, the measured effective length, λ_S, can be directly related to the λ_{pin} and the flux flow resistivity [9]-[10]. However, how these lengths are related to each other quantitatively can be specified by a direct calibration of the setup [8]. The method of calibration of the coil's field configuration with normal conducting copper plates, we realized, enables correct transferring of the measured shifts in frequency, δF, to the changes in distance from the coil, d. We made it by moving different diameter ($\Phi = 2R$) disk-shaped thin copper plates towards the coil face up-to the given distance, d_o, and back. This changes the field configuration and permits to get the geometric G-factor as the coefficient for the resonant frequency (coil inductance) modulation. Changing the position of the metallic object, one can determine the value of the G-factor as the relation between the frequency modulation δF and the change in position δd (Fig.2a).

Figures 2-3 illustrate the results of the calibration for the used technique. It is seen how the determined $G(d_o,S)$-factor depends on the sample's area, S, and position, d_o, near coil. In fact, the G-factor determines the resolution of the technique. It allows to transfer the measured shifts in frequency to the changes in distance by: $\delta d = -G(d_o,S) \cdot \delta F$ (in particular, to the real part of the λ by $\delta\lambda = -G(d_o,S) \cdot \delta F$, important for the extraction of the material parameters in HTS). Fig.2 shows exponential drop of the sensitivity of our technique with a distance due to decrease of the field's density. $G_w \sim 0.9$ Å/Hz is typical value of a geometric factor achieved at the optimal workpoint. The high stability of our TD oscillator (typically, $\Delta F_{stab} \sim 1$-3Hz at $F_{meas} = 23$MHz [2]) enabled to reach the resolution of about $\delta d \sim \delta\lambda_S = G_w \cdot \Delta F_{stab} \sim 1$-3Å in our device.

Figure 3 allows to estimate the optimal cross-sectional size for the samples. For such samples (1mm<R<3mm) the frequency shift is proportional to the area ($\delta F/S$=const). Fig.3 shows also the field configuration near coil, which is almost similar to the calculated diagram of the field lines presented along the X-axis, for comparison. Fig.3 illustrates also the imperfections of our hand-made coil design (~1mm-size hole in its center), causing sharp decrease of the sensitivity for small samples (R<1mm), because of distortion of the field lines near coil center. But, for the used 5mm disk-shaped thin film the main mistakes at the frequency tests (caused by the field inhomogeneity) are less than $(1mm)^2/(5mm)^2 = 1/25 \sim 4\%$ due to hole in the coil center and less than 7% because of the field inhomogeneity on the rest parts of the sample (see the line d_o=1400μm in Fig.3). It is small enough to be neglected in this study. In [8] we discuss ways how one can overcome this problem and make the technique and the results better.

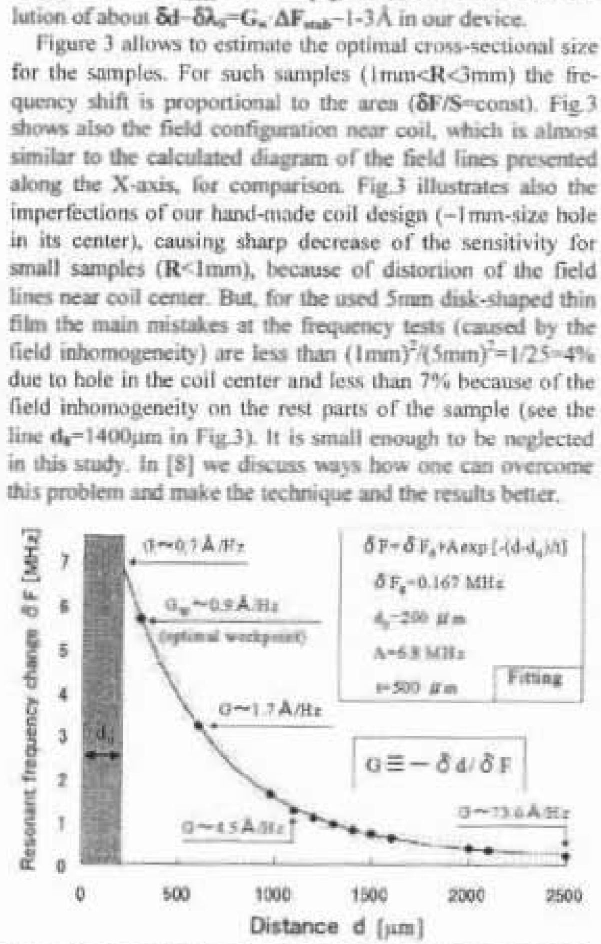

Fig.2a. The flat coil's G-factor (resolution of the technique) vs. the position (δF vs. distance, d) for Φ=7.25mm thin copper plate (curve No.9 in Fig.2b).

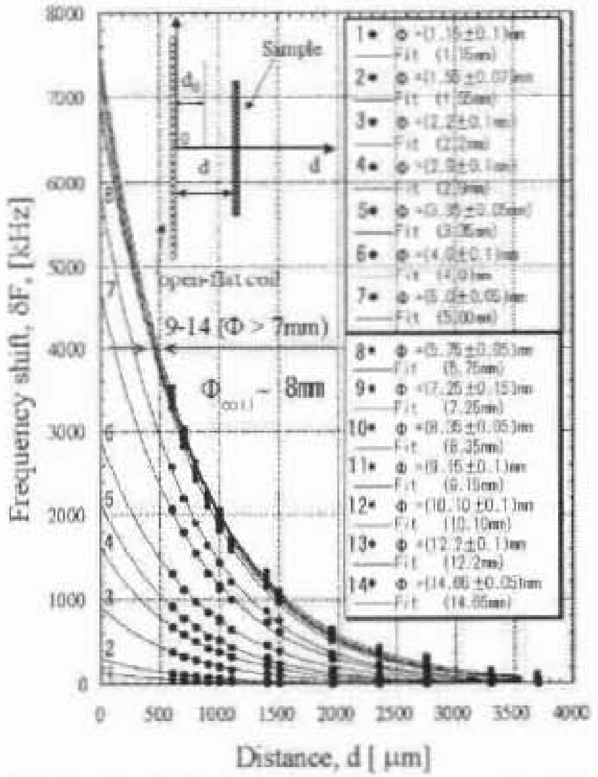

Fig.2b. The flat coil's Q-factor (resolution of the technique) vs. the position (δF vs. distance, d) for different diameter (Φ=2R) thin copper plates.

III. Results and Discussion

Below, we present the results of the study of the magnetic transition curves in Y-Ba-Cu-O thin film detected by the frequency and amplitude of the F_{meas}≈23MHz TD oscillator simultaneously. The Φ=5mm disk-shaped sample was patterned by the chemical etching method from the c-axis oriented and 0.2μm thick film [11]. The data enabled to construct the magnetic phase diagram for the tested small-volume Y-Ba-Cu-O thin film. During the tests by this technique we could detect an anomalous absorption of the testing RF field's power by a film close to the end of its magnetic S/N transition.

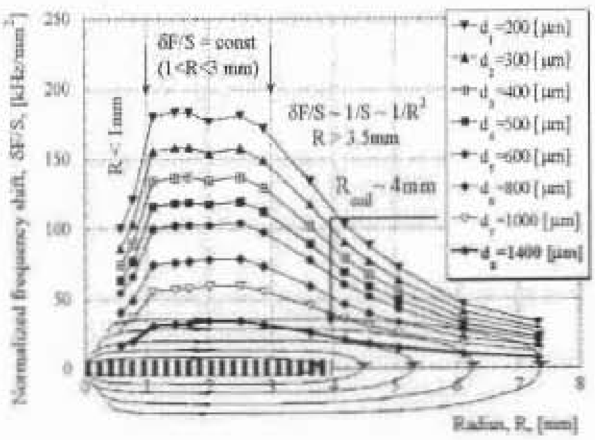

Fig.3. The normalized sensitivity (δF/S) vs. the copper plate cross-sectional sizes (R and S are the sample's radius and area) for various distances from the coil, d_n, as a parameter. d_8=1400μm is the tested HTS film's position.

Typical curves for δF, $\delta\lambda_S$ and δA vs. **H**(||c) at different temperatures are shown in Figs.4-6. First of all the figures demonstrate that the technique operates in up to 12T fields, measures the changes of the shielding length in a range ~1mm and shows 10^{-4}–10^{-3} resolution (depending on film's size), which is superior to SQUID's. It enables to detect the changes ~10^{-8}W of a power absorption by a film, originated from the variation of the flux distribution inside it, which is superior to SQUID's too. Besides, our technique has an excellent resolution near T_c, which is due to the flat shape of the coil.

The Figs. 4-6 show also that the frequency transition curves are finally saturated at the same constant value for high enough **H**. The frequency after the saturation corresponds to its value in the normal state (to an accuracy of the oscillator parameters' temperature dependence). The background drift in frequency and amplitude over the duration of the tests was negligible. Besides, no magnetic hysteresis was observed at the further decreasing of the field at any tested temperature (provided that the temperature is stabile enough). Thus, one can determine the upper critical field by the field value at the saturation point. Fig.7 shows the $H_{C2}(T)$ curve (●). It allows to determine the T_c=88.7K and the value of $dH_{C2}/dT(T_c)$= −0.73 ±0.02T/K by a linear extrapolation to T_c of the $H_{C2}(T)$. The $H_{C2}(T)$ data obey the $H_{C2}(0)\cdot[1-(T/T_c)^2]^\beta$ dependence with the $H_{C2}(0)$= (69.5 ±1.5)T and β=1.26 ±0.02.

We could also estimate the line, $H^*(T)$, corresponding to the percolation limit for the unpinned clusters, (◆). The inset in Fig.6 illustrates the method of determining of the H^* by the onset point of a deviation from the linear dependence of the amplitude curve at small fields. Seems the $H^*(T)$ line is almost identical to the depinning line (■), $H_{dep}^{transp}(T)$, determined by the 4-probe transport test method using similar quality 1mm-long and 200μm-wide bridge [12] (which is equal to the vortex Glass-Liquid transition line, $H_{GL}(T)$). The $H^*(T)$ data obey the $H^*(0)\cdot[1-(T/T_c)^2]^\delta$ dependence too with the $H^*(0)$= 30.8 ±1.1T and δ=1.49 ±0.03 close to T_c.

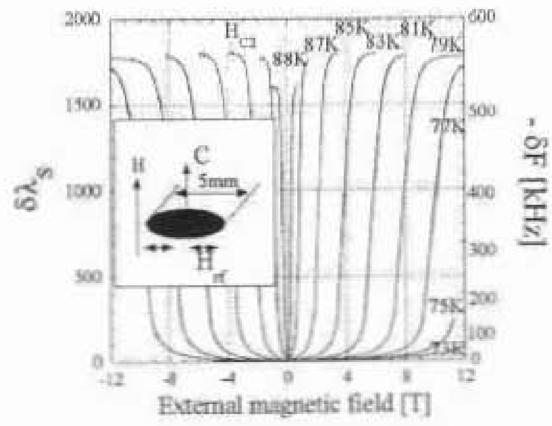

Fig.4. Frequency magnetic transition curves. Inset – field configuration.

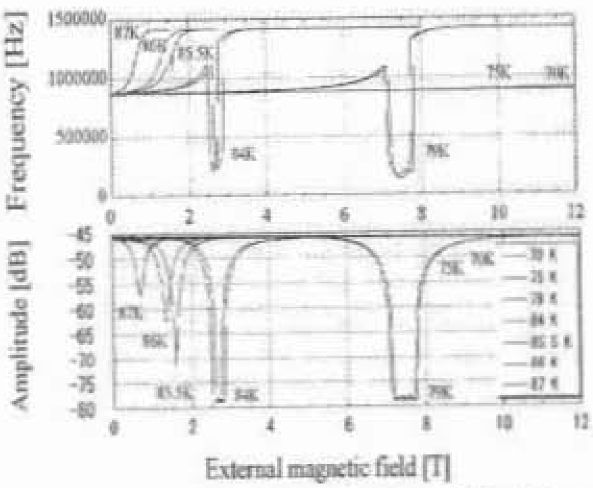

Fig.5: Magnetic transition curves (distance from the coil - 1400μm). The peculiarities on the frequency curves (top graph) relate with the absorption of testing field's power by a sample at the fields, H_{abs} corresponding to the respective peaks of absorption shown on the amplitude curves (bottom graph).

By this technique we detected also an anomalous absorption of the testing field's power by a film close to the end of its S/N transition (▼), $H_{abs}(T)$, which lays between the $H^*(T)$ and $H_{C2}(T)$ lines on the phase diagram shown in Fig.7. The peculiarities registered on the frequency transition curves at H_{abs} (Figs.5-6) are also due to the absorption, because of the known dependence of the circuit's resonant frequency on its Q-factor by $F = 1/(2\pi\sqrt{LC})\bullet[1-\omega_0 L/(Q\bullet|R_n|)+\cdots]$ [7]. Here R_n is TD's negative-differential resistance and $\omega_0=1/\sqrt{LC}$. What we could detect is not a device effect. The similar effect was detected also in Y-Ba-Cu-O crystal [13]. It becomes noticeable when the absorption is large. Moving the specimen far from the coil or testing small samples, and also if the oscillator is tuned far from its steady operation threshold, one can detect the clean (without peculiarities) frequency transition curves.

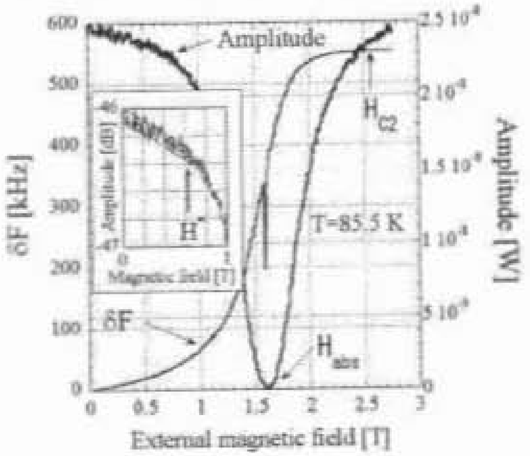

Fig.6: Transition curves at 85.5K (film's distance from the coil - 1400μm): The peculiarity on the δF(H) curve at H_{abs} corresponds to the absorption of testing field's power by a sample. Inset - determining of the H^* by the onset point of a deviation from the linear dependence of A(H) at small fields.

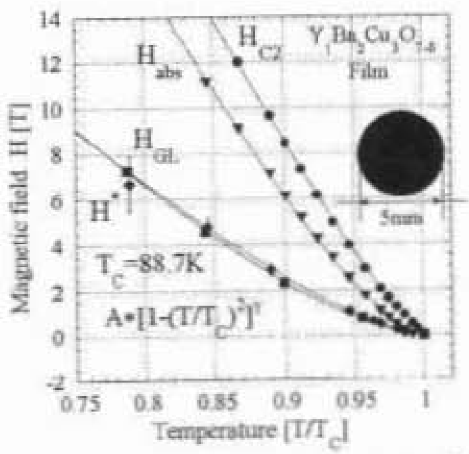

Fig.7: Magnetic phase diagram: Temperature dependencies of the upper critical field (●), $H_{C2}(T)$, the depinning field related with the unpinned clusters (◆), $H^*(T)$, and the field value (▼), $H_{abs}(T)$, related with a detected absorption of the testing field's power by a film. The data for the vortex Glass-Liquid (GL) phase transition line (■), $H_{GL}(T)$, are taken from [12].

However, the final understanding of the nature and the reasons of such an anomalous absorption need additional study. This technique could detect the effect, because the method, due to its specific flat design has advantages and is irreplaceable at studies with small-volume samples, especially near T_c.

In conclusion, we developed a flat coil-based testing technique and applied it for studies of vortex dynamics in HTS. It strongly improves the abilities of the traditional "LC resonator" method, especially at tests in small-volume samples with a small signal. It is also an excellent RF Q-meter for the detection of small changes of absorption in HTS. Besides, our technique is irreplaceable in sensitivity especially at tests near T_c. Some of its capabilities are superior to SQUID's. These allow use it to study fine peculiarities of the superconductive state in small-volume HTS at the beginning of its formation, probably, unnoticeable for the other techniques. Besides, the simultaneous frequency and amplitude measurements allow to construct the magnetic phase diagram in thin-film HTS. This is important for many applications, because there are no undisturbed testing methods at present, especially sensitive near T_c, which can construct the magnetic phase diagram for small-volume HTS during the same and simple measurement.

REFERENCES

[1] A. Maeda and T. Hanaguri, *Supercond. Review*, vol. **3**, 1, 1998.
[2] S.G. Gevorgyan, T. Kiss et al., *Rev. Sci. Instrum.*, vol. **71**, 1488, 2000.
[3] Y. Sharvin and V. Gantmakher, *J Exp Theor Phys*, vol. **39**, 1242, 1960
[4] S. Patnaik, R.C. Budhani et al., *Physica C*, vol. **309**, 221, 1998.
[5] C.T. Van Degrift and D.P. Love, *Rev. Sci. Instrum.*, vol. **52**, 712, 1981.
[6] S.G. Gevorgyan, A.A. Movsisyan, G.D. Movsesyan, V.A. Shindyan, and H.G. Shirinyan, *Mod. Phys. Lett. B*, vol. **11**, 1123, 1997
[7] S.G. Gevorgyan et al., *Rev. Sci. Instrum.*, vol. **69**, 2550, 1998.
[8] S.G. Gevorgyan, T. Kiss et al., *Rev. Sci. Instrum.* (to be submitted).
[9] E.H. Brandt, Phys. Rev. Lett. , vol. **67**, 2219, 1991.
[10] M.W. Coffey and J.R. Clem, Phys. Rev. B **45**, 9872, 1992.
[11] T. Nakamura et al., *Inst. Phys. Conf.*, Ser. 158, 1017, 1997.
[12] T. Kiss, et al., *Supercond. Sci. and Techonology*, vol. **12**, 1079, 1999.
[13] S.G. Gevorgyan et al., Proc. of the 4-th Workshop on Applied Superconductivity in Kyushu Area, Fukuoka, Japan, 1999, pp. 57-60.