One of the Way of Tc Detection for the Unidentified High-Tc Superconductor

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Abstract :
There has been considerable controversy over the measured points of superconducting transition temperature (Tc) since especially in 1986 . While, the devices operating above the boiling temperature of liquid nitrogen are expected to be more attractive than low temperature superconducting devices because of the great importance for the utility industry. In this paper, I would like to introduce the new way of Tc detection for the unidentified high-Tc superconductor into the discussion of this field.

Ⅰ. Introduction

First of all, upon the detection of the Tc, it is necessary for me to produce the unidentified high-Tc superconductors. Therefore, I would like to bring up the copper-oxide-based one which were heat-treated by myself as the samples of the trial products in this research. Among of these copper-oxide-based high-Tc superconductors, the heat-treated one in 1997 seems to behave like the highest-Tc superconductor in particula. Nowadays, after much of three years later, it still make the Meissner effect signal definitely, and contrary to general belief, the results of the experiments say that the present Tc seems to be higher than the former Tc. Though these are the results which have a peculiar appeal, the way of Tc detection for the high-Tc superconductors require the very careful handling indeed. So, after the producing unidentified high-Tc superconductors (namely, copper-oxide-based one), I would like to illustrate the Tc detection of the used way here in detail.

Ⅱ. Producing the Samples

As mentioned above, I would like to define the unidentified high-Tc superconductors as the sintered conductors which the Tc are not clear. Therefore, even if the conductors are Y based copper oxides known for well as the high-Tc one, I would like to give the definitions of the word unidentified high-Tc superconductors, because the Tc has not yet been precisely cleared since the Y based sintered conductors were first made at the study room of Nagoya institute of technology (shortened N. I. T) in 1997. Then it is my turn to explain the way how the Y-based sintered conductors were produced in this study. So the first thing we had better do is generally to get the chemical elements of materials for producing high-Tc superconductors, and so did I. After the events, the rest of the way of the making one is as follows.

(1) Mixing the chemical elements of materials,
(2) Shaping of the samples,
(3) The preliminary sintering,
(4) Real sintering.

In these circumstances, mixing the chemical elements of materials is the first process of making sintered conductor and is experimentally carried out as planned. That is, the pestle and mortar are employed in this experimental early stage here too. And, the chemical elements of materials are essential to product the unidentified high-Tc superconductors.
Therefore, "what kinds of the chemical elements should I employ" is the significant issues for the results of this study. After all, I used the powdered BYCO elements as the materials of the superconductors and got them from HAYASHI chemical industrial Co, Ltd of KYOTO in JAPAN because the powder is suitable one for the reasons of given below:

(1) The BYCO compounds are well known for the high quality of superconductivity.
(2) There seem to be the significant differences of Tc (superconducting transition temperature) values depending on the methods of sintering treatment.
(3) I may be able to make the trial products as the block BYCO compounds, that is, the samples are the bulk materials in this study.

And, the powder has an average diameter of 1.99 (μm) in the HAYASHI's view based on the experimental results. Furthermore, the HAYASHI's view says that the sintered conductors made from this powder behaves like a superconductor within the limits of Tc -183 (℃), if the sintering treatment is well carried out with the moderate oxygen in the smelting furnace. And then the descriptions of the elements sample say that the sintering treatment should be done on the two necessary conditions, namely, the temperature of real sintering is 810 (℃) and the time needed for the sintering treatment is about 10 hours.

Ⅲ. The Samples

Mixing the chemical elements of materials is not troublesome procedure because of its small amount of chemicals in this study. However that may be, the sufficient work of mixing it up is not easy task to product the good samples. What does the word 'good sample' mean in this case? There may be some readers who just open to such a question, I guess. So obviously, if we find ourselves in a agreement at the sense of the word 'good sample', you will get one. There, I would like to give the definition of the word 'good sample' as the conductors having the desirable superconductivity for the significant technological applications. While, the conductors displaying the low superconducting transition temperature are given the definitions of poor quality one, especially in this paper. Both of them however are given the definitions of the unidentified superconductors at the stage of just over the real sintering respectively. Because it is still hard to make clear the distinction between the good samples and the poor samples at that stage. This means that it is still hard to get the superconducting transition temperature (Tc) theoretically, and suggests that not only the processig but also the method for making sure of the properties is essential to get the unidentified good samples. Therefore, the K_1 type superconductors[1] which I got in 1997 will be used mainly as the samples of unidentified one in this paper because of making sure the two items for the reasons given below.

(1) Even now after much of 4 years, does the K_1 type one work as the superconductors?
(2) Judging from the course of things so far, there is a possibility of changing into another one as time passed.

Ⅳ. The depository of the K_1 type superconductors

I have been safekeeping the K_1 type superconductors in the desiccator making dry one with desiccant sillica gel since the samples were produced in 1997. From '97 to 2000 that is, during the three years, K_1 type one had been working as the samples of high-Tc superconductors (see the reference[2]). But I wonder if the K_1 type one will work as well as the checked one of the last year even in nowadays. The desiccator is a general-purpose one and is constantly keep the proper environment of the inside area for the samples of super conductors including K_1 type one. Now, there is no way of returning, so my mind is already in checking the aging of high-Tc superconductors. Therefore I think the first thing I'd better do is to check again the properties of K_1 type one following last time[2] in 2000. But there is a catch to it, that is, I have to get the correct Tc as a matter of course and this is not so easy because the samples become superconductors just suddenly and instantaneously at the quite low temperature in general. So what I suggest is making use of the computer aided thermistor system to get the realistic and correctly Tc. Needless to say, we should make an experiment with the computer aided thermistor system and should make sure if it's possible or not to get the realistic Tc. By the way, the analog-digital converter that will impart desirable properties and shapes is necessary for this research, and if it's possible to get such converter, I seem to be able to make use of the lower costed measuring instrument[3] as an example of the way to check the Tc.

V. Measurement of the Tc

On the occasion of the measurement of the Tc, it is necessary to postulate the range of valid temperature and is necessary to set up the output voltage within the limits of 5 【V】 at the same time. So I hope to set up that the low temperature is the boiling point of liquid nitrogen and the high temperature is the room one, namely they are about (−) 196 (℃) and 27 (℃) respectivery. And then, these numerical values have to be filled in the form of the sensor file which the manual[4] says as a matter of fact. Furthermore, the components of the computer aided thermistor system such as thermocouple, amplifier, and connectible lead (this lead also known as a modular cord) are all necessary to construct the good system. Among these components, the thermocouple plays an important part in the checking the temperature around the surface of the K_1 type samples. Because the range of valid temperature to detect is extremely at low, the thermocouple made a pair of copper and constantan can be applied to this case, so the first component to construct the system is selected with ease and the thermocouple which I've been puting to use is the standardized goods [0.65 × 1P T 1000] of JIS. In this representation with a symbol, each means the wire's diameter of 0.65 (mm), one pair, the couple of copper and constantan, the wire 1000 (mm) long, respectively. And the catalogue says that the thermocouple made a pair of copper and constantan works with the error of less than ±1 (℃) or ±1.5 (%) in the low temperature measurement ranging from 0 (℃) to (−) 200 (℃). So, if the measured value times ±1.5 (%) is larger than ±1 (℃), the tolerance is supposed to be less than ±1.5 (%) by the contents of the catalogue. Next, though the second component to construct the system is the amplifier and is also significant part because the electromotive force generated by the difference in the temperature is little or tiny as a matter of fact. According to what I tested, the output of the thermocouple pairing of copper and constantan was about 6.00 (mv) at best even if the sensor was working well in the liquid nitrogen. And, when the sensor was in the room temperature at 20.6 (℃), it was about 0.83 (mv). These values of electromotive force are so small that we will not be able to put them to good use for the temperature measurement, that is, the proper amplifier is necessary for this job. Therefore, the amplifier has to be selected in accordance with the criteria for selection such as the analog output 0～5 (V) and the range of temperature measurement (−) 200 ～ (+) 50 (℃) in particular. Considering the circumstances mentioned above, I've been using the amplifier made by company on the recommendation of an amp-sales agent and it seems to work well as far as that goes. After the amplification, the signal voltage of temperature measurement is put into the analog to digital converter as an incoming signal with the connectible lead known as a modular cord. Here, the modular cords which consists of four conducting wires are generally on the market, so we can easily get them if we wish as a matter of course, and so did I. Then, the incoming signal at the entrance of the A/D converter[3], it is analogical signal named the voltage, but if it once comes out of the exit, it is converted into the digital one to make a computeranalysis of the behavior of superconducting transition temperature. So the quality of the analyzed results depends on the data of the programing such as 5(V)/8191/50(℃), 0(V)/0/(-)200(℃), and 0(V)/(−) 8191/(−)200(℃). Here, the 5(V) is the maximum output of the sensor (i.e the output of thermocouple), and means that the output voltage 5.00(V) to 0.00(V) is divided into 8192 (i.e 13 bit). Further the 8191 is the value of binary digit indicating the highest temperature in the range of this measurement. These data are acceptable one in general but there is an opinion of the converter designer[3] about the maximum output 5(V) of the sensorvoltage, that is, he is of the opinion that we should not fix over the full-voltage of 5(V) and should fix one less than 5(V) to get the accuracy of temperature measurement. These are the matters that require attention to the measurement of superconducting transition temperature, while the another program is necessary for the observations of the resistivity and is set up as follows, namely, 5(V)/8191/100(V), 0(V)/0/0(V), and 0(V)/(-)8191/(-)100(V). However, as we should investigate the behavior of superconducting transition resistivity within the limits of one at the room temperature or under, so the program above ought to be modified as follows, 5(V) / 8191 / (-)0.00(V), 0(V) / 0 / 0(V), and 0(V) / (-)8191 /(-)1.00(V) because of the main two reasons. One of them is that the resistivity takes on a different

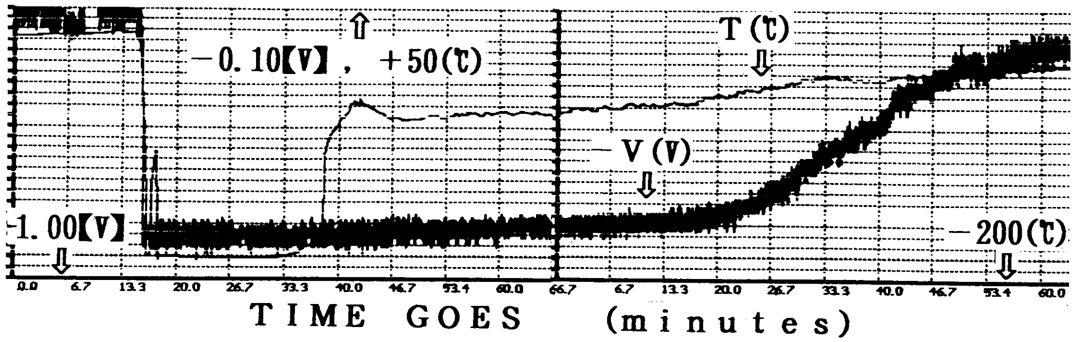

Fig. 1, After the liquid nitrogen evaporated, We can get the appropriate environmental temperature to find the Tc.

character from its voltage as if they are in the opposite vectors each. Therefore, when we check up on the behavior of the resistivity, we should give attention to the behavior of the negative voltage generating with the electrical resistance. Further, the other reason for modifying the above program is that the values of the superconducting transition voltage is a little bit as an absolute quantity in the first place. And, we can even eatimate the values of maximum voltage at the beginning of the experiment if we obtain the values of the electric current and resistance at the room temperature. In this experiment, that is why 1.00(V) is a good enough for the values of maximum voltage. This means that we will be easily able to set up the range of voltage and to start the experiment on the detection of the Tc in the state of temperature dropping from room one to boilling point of liquid nitrogen (≒(−) 196(℃)). Shown here in Figure 1 is one of the experimental results by the above program. Where, the bold line represents the negative voltage generated with the resistance of the samples, and the thin line indicates the temperature nearby the samples. This is just the first result I've been looking for in this research, and shows that these two factors are correlated definitely. That is, Figure. 1 represents the correlation between negative voltage and superconducting transition temperature or represents the correlation between superconducting transition resistivity and superconducting transition temperature. The latter one is the center focus of public attention and is noteworthy phenomenon for this reserch, too.

Ⅵ. Some Data Based on This Experiment

All right, let's investigate the Tc of the unidentified high-Tc superconductors on the figure 1. In the first place, I fitted the sample as a circuit element on the measuring instrument, where the resistance value of the sample is about 46 [Ω] in the room temperature. And then, I changed it into the resistor 46 [Ω] , 50 [Ω] in turn. Therefore, We can find these reasonable traces at the biginning of the correlation between the resistivity and the temperature. Among of these four bold clusters, the 1st and 4th one are the traces of the same unidentified high-Tc superconductors, namely, the 4th bold line is the center of our attention after 13.3 minutes from the start in particular. A few minutes later, the 4th bold one, namely, the resistance value of the unidentified high-Tc superconductors drops sharply into the pattern of rapid cooling because I just poured liquid nitrogen into the vessel. Then the vessel is almost filled with the liquid nitrogen, the unidentified high-Tc superconductors, namely, the sample is covered with the liquid nitrogen as a matter of course. Therefore, if the sample is sure the high-Tc superconductors, the Tc must be exist on the way of cooling to the boilling point of liquid nitrogen, but it is hard for me to catch or confirm the Tc because the cooling will pass by at tremendous speed and will reach the degree of about (−)196 (℃). We can observe the states above within the limits of 20.0 minutes. Then the temperature once reached the lowest one, the negative voltage also get lowest as we can see in the range of about 20.0 to 33.3 (minutes). Under this circumstances, these behavior suggest that the sample is sending the Meissner effect signal even if it is covered with liquid nitrogen. So we must be able to find out if the magnet floats on the sample or not in the liquid nitrogen. According to the conclusion based on the experimental results, I have no doubt that the magnet floats even if it sank in the deep liquid nitrogen. Then, a few minutes later, we can see the

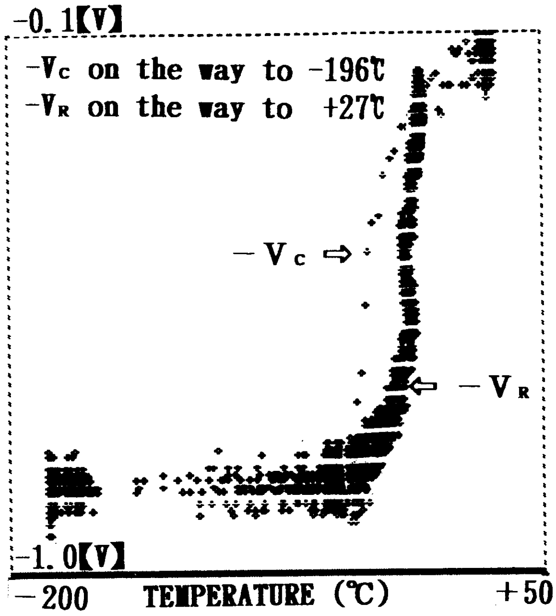

Fig. 2, By taking notice of this correlation, I gradually got the general idea of what had happened. But the Tc is still unclear.

temperature rises quickly with the vaporization of liquid nitrogen and leaving it in the air. But, the other side, the negative voltage has been kept intact as if it stands at the state of the zero resistance. This means that the sample is still in good condition to be a superconductor even if the temperature in the vicinity of the sample rises quickly to about (−)40(℃). As a matter of course, the Tc of this sample seems to be extraordinarily high as far as the eye could see on the Figure.1. So I think the next thing we had better do is to make a graph of the correlation between the negative voltage and the temperature in the vicinity of the sample. Figure. 2 is indeed the suitable representation for it and represents the behavior of correlation between the negative voltage and the temperature in the whole area of the passage of time. As we can see in the Figure. 2, the correlation between the negative voltage and the temperature seems to indicates the representative superconductivity of the sample, and seems to indicates the noteworthy phenomenon in this case. Namely, I can see the hysteresial phenomenon on this behavior of correlation during the temperature dropping and rising process. This means that we may be able to witness the magnet falling down to the sample at a slow speed. And, if it is possible to do so, we may be able to know the temperature at that time by means of checking one against the data kept on the computer applications. As you know, such a temperature is applicable to the Tc in this study because it is major Tc for practical use. Therefore, the distance between the sample and the measuring point of the temperature is a significant factor in the success of this study. So, I must repeat that the measuring point mentioned above does not touch the sample but is a bit (about 1 (mm)) away from the sample. Namely, I hope you can accept that the way of Tc detection mentioned here stands for the method of Tc detection under the conditions of that the measuring point of the temperature is in the vicinity of the sample. By the way, even if we obtain the items mentioned above, it is still hard to get the reliable Tc because of insufficient evidence. "That is, the Meissnereffect signal and the time required for the magnet falling to the sample from the experiments started are essential to get the reliable Tc". In these two items, what is particularly important is latter one because the data corresponds to the Tc is not fixed if the time is unclear. Therefore, I can't find out how many degrees of the reliable Tc is in the figure. 2, because I didn't get the time at the instant when the magnet fell to the sample as a matter of fact. So, taking care that I don't miss the time at the instant, I made the experiments with same sample over and over again. Then the Figure. 3, and 4 show the some results of the experiment. So, as we can see in the Figure. 3, the arrow at the abscissa 33.3 (minutes) indicates the behavior of the negative voltage and the temperature at that instant just as the floating magnet almost fall to the sample. Figure. 5 is a photo taken at that time by myself and the magnet seems to be floating still. After I finished taking the photo, the landing of the floating magnet was confirmed, that is, the magnet was on the surface of the frozen sample. It was about 34.3 minutes later than the biginning of the measurement. By the way, as the measurement is supposed to conduct at a speed of once a second[3], it is possible to find out the Tc in the saved data. For example, the data at 33.3 minutes later than the biginning of the measurement corresponds to the 1998th (∵ 33.3 times 60) value of the measurement, thus, the coordinates of the 1998th dot are found out such as (temperature, negative voltage) = (−91.02 ℃, −0.74 V) in the saved data. And, since the data at 34.3 minutes is treated in the same way, the coordinates of the 2058th dot are found out such as (T, −E) = (−89.01 ℃,

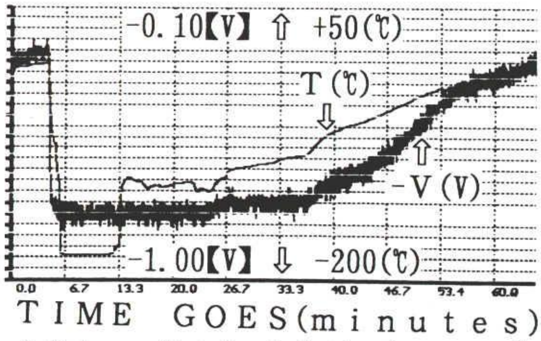

Fig. 3, Meissner effect signal, that is, the magnet floating in the air was seen with my own eyes at abscissa 33.3 minutes. (Fig 5 is a photo taken at that time.)

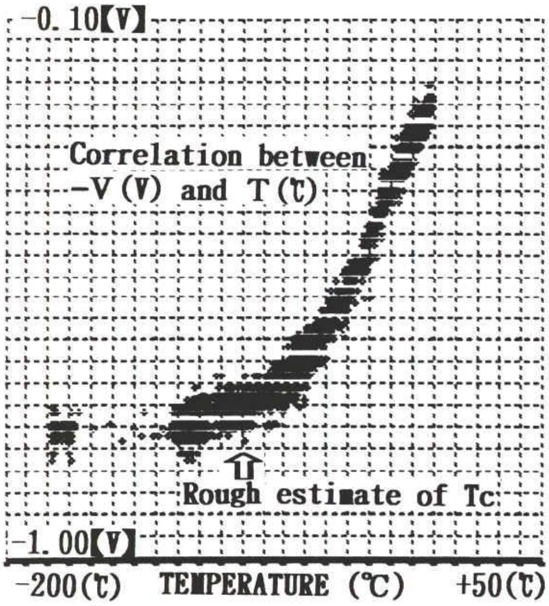

Fig. 4, I may be able to obtain the Tc if I know the time on the fall of magnet.

−0.75 V) in the saved data. These two points of coordinates mean that the superconductible critical temperature of this sample is about −91.02 (℃) even if they are in the bad position. This value of the Tc is significant one for us because the world's highest Tc is still now about (−) 103 (℃)[4] as far as I know, however, since the acquired Tc in july of last year was about −97 (℃)[2], it is necessary for me to check up on the difference between −91.02 (℃) and −97 (℃).

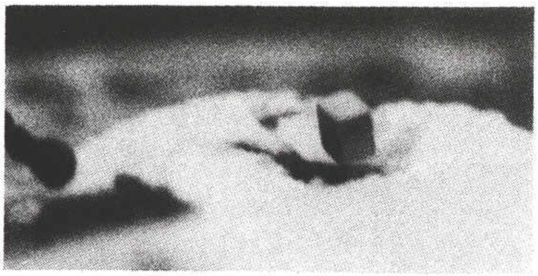

Fig. 5, The magnet just before the fall. About one minute later, the magnet fell on the sample and gave a hint to get the Tc.

Ⅶ. Conclusions

According to the results of this Tc detection mentioned here, the value of detected one seem to be an unusually high, so, it is necessary for me to check the accuracy of the measurements and to check the difference between −91.02 (℃) and −97 (℃) with dependable way, however, as to the detection of the Tc above mentioned, it is possible with ease to witness the instant of the magnet falling to the upper surface of the sample because the magnet floating in the air falls slowly just at that moment. This means that the human error in this observation is small and the −91.02 (℃) is a noteworthy value as the critical temperature of unidentified high-Tc superconductor.

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