Stability of multi-core MgB_2/Ti/Cu/SS wires.

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Abstract

Stability of 19-filaments MgB_2/Ti/Cu/SS wires carrying the transport currents has been examined. Properties of two identical wires annealed at 600 ^oC/2.5 h and 800 ^oC/0.5 h were compared. It was found that annealing conditions influence not only the current carrying capacity of MgB_2 filaments but also affect the inter-diffusion at Ti/Cu and Cu/SS interfaces, which is worsening the electrical and thermal conductivities of composite elements. Consequently, differences in wire's resistances and *I-V* quenches were measured, which are correlating well with experimentally estimated minimum quench energy and normal zone propagation velocity.

Keywords: MgB_2 wire, resistance, *I-V* curves, transport currents, *n*-exponents, minimum quench energy, normal zone propagation velocity

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1. Introduction

The occurrence of a normal conducting zone and the following heating effect can cause a permanent damage of any superconducting magnet. It depends on the conductor's thermal properties and on operating conditions (current, magnetic field and temperature) [1]. Thermal stability is related to the maximum current that a superconducting filament can carry. Usually, the minimum quench energy (MQE) and the normal zone propagation velocity (v_p) are analysed experimentally, or predicted with the aid of analytical formulae [1-2]. MQE is the amount of heat which is needed to trigger the avalanche effect and v_p says how fast a normal conducting zone grows along the conductor [3]. Thermal avalanches of this type can be

stopped by stabilising the conductor, either by offering low-resistance paths around the hot-spot or by making the filaments thinner and cooling more efficient. Therefore, practical and stable MgB_2 wire needs to be fine-filamentary structured and also thermally stabilized by high conductive metallic element. Oxygen free high conductive copper (OFHC) is a widely used material for stabilized technical superconductors and it was already applied for MgB_2 wires [4-5]. Stabilizing copper reacts strongly with magnesium at high temperatures and therefore has to be carefully protected by diffusion barrier (e.g. Nb or Ti). Inter-diffusion was observed at Ti/Cu and NbTi/Cu interface of MgB_2 wires [6-8]. The strongest interface and temperature dependent interaction have been observed between the copper stabilizer and titanium barrier. Latif have shown five possible inter-metallic layers (Ti_2Cu, TiCu, Ti_3Cu_4, Ti_2Cu_3 and $TiCu_4$) created between Ti and Cu during annealing [7]. Contamination of stabilizing Cu is worsening the electrical and thermal conductivity, which is worsening the thermal stabilization. Therefore, detailed studies of stability throughout the temperature-field domain have to be done for MgB_2 conductors containing different metallic barriers and copper.

This paper presents characteristics focussed to the stability of 19 filaments MgB_2 wire heat treated at two different regimes 600 ^oC/2.5 h and 800 ^oC/0.5 h.

2. Experimental

A 19-filaments wire consisting of 50% SS-reinforcement, 19.5% Cu-stabilization, 19% Ti-barrier and 8.5% MgB_2-superconductor has been made by *in-situ* approach [9]. The wire was drawn to the final diameters of 0.86 mm and finally heat treated (HT) at two different conditions: 600 ^oC/2.5 h and 800 ^oC/0.5 h in pure Ar atmosphere. Optical microscopy (OM) of wires crosssections slightly etched by HNO_3 acid solution was used for characterization of composite structure after annealing. Fig. 1 shows the cross-section of the wire observed by optical microscopy after HT at 600 ºC/2.5h. The temperature dependence of both wire's (800 ^oC and 600 ^oC) resistances, $R(T)$ between 30 and 300 K, and the dependence of critical current, $I_c(T)$ between 22 and 32 K, have been measured at self-field and in vacuum using a cryocooler. Critical currents (I_c at 1 μV/cm) were measured also at 4.2 K in the external field ranging from 3.5 to 12 T. Standard current-voltage (I-V) measurement for $I >> I_c$ have been done with the aim to analyze the thermal stability of both wires. The constant transport current ramping rate α_{up} = 0.33 As^{-1} and α_{down} = -0.33 As_{-1} was applied [10]. The quench development of MgB_2 conductors has been analysed experimentally. Energy pulses were deposited to the conductor by passing rectangular current pulses through a graphite-based-epoxy heater. The length of the heater is 2 mm and its resistance about 100 Ω. In order to

calculate the MQE with precision, the voltage across the heater and the current passing through the heater are measured in each experiment. The electric field profiles around the point heat disturbance that gives rise to a quench, as well as their time evolution, were measured from multiple voltage taps along the conductor using a Data Acquisition (DAQ) device. The measurements have been done at self-field and in vacuum (adiabatic conditions) using a cryocooler. The sample was thermally anchored to the 2nd stage of the cold head of the cryocooler. The temperature of the sample can be varied from 11 K to > 40 K using a Lakeshore 331 temperature controller with a stable thermal environment suitable for quench measurement at transport current up to 140 A [11]. The heat pulse for the onset of irreversible growth of the normal zone was used to define the minimum quench energy (MQE). The propagation velocity (v_p) was obtained directly from the growth rate of the normal zone.

3. Results and discussion

3.1. Wire's resistance

Figure 2a shows the resistances of short wire samples (0.86 mm in diameter and 20 mm in length) measured between 30 and 300 K. It is apparent that the wire heat treated at 800 ^{o}C has much higher resistance. Considerably different residual resistivity ratios were obtained for these wires: RRR_{800} = 3.42 and RRR_{600} = 7.94. The ratio of both wire's resistance at 40 K is R_{800}(40 K)/R_{600}(40 K) = 6.35 (R_{600}(40 K) = 0.057 mΩ, R_{800}(40 K) = 3.62 mΩ) and at room temperature R_{800}(300 K)/R_{600}(300 K) = 2.84. These resistance differences have to be caused by different diffusion/reaction between the composite elements at applied heat treatment conditions (600 ^{o}C/2.5 h and 800 ^{o}C/0.5 h). It was already shown that especially strong interaction occurs between the copper stabilizer and titanium barrier, which is strongly temperature dependent [7, 12]. EDX element distribution confirms smaller reaction at Ti/Cu interface when annealing temperatures was decreased from 800 to 600 ◦C [12]. While the optical microscopy does not reveal any interaction between SS and copper (see Fig. 1), the temperature-dependent layer SS−Cu has been found by EDX [12]. All mentioned inter-diffusions are worsening the electrical conductivity of composite elements and consequently increase the wire's resistance, see Fig. 2a.

Critical temperatures estimated as a middle of resistive transition $T_{c\text{-}600}$ = 32.07 K ($\Delta T_{c\text{-}600}$ = 3.29 K) and $T_{c\text{-}800}$ = 34.55 K ($\Delta T_{c\text{-}800}$ = 2.33 K) were obtained, see Fig. 2b. Lower critical temperature and wider transition are attributed to solid-state reaction of Mg with B at 600 ^{o}C, where MgB_2 phase is created through inter-diffusion mechanism. Above the melting point of

magnesium (≈ 649 ^{o}C) the solid-liquid reaction takes place influencing the kinetics of MgB_2 phase creation leading to different stoichiometry and morphology than for solid-state. The complete MgB_2 phase creation is formed when sintering temperature exceeds to 750 ^{o}C/1h [13]. It was already shown that T_c is systematically increasing with heat treatment temperature [14-15].

3.2. Critical current densities

Figure 3a shows the effect of final annealing on $J_c(B)$ characteristics measured at 4.2 K. Higher J_c were obtained for 800 ^{o}C wire in whole field region, but higher transport current density (≈ 16 000 Acm^{-2}) was possible measure in low field region (B < 4 T) without quenching for 600 ^{o}C wire due to its better stability. It was not possible to measure J_c > 12 800 Acm^{-2} in the wire heat treated at 800 ^{o}C (at B < 4 T). The insert in Fig. 3a shows n-exponents of both wires correlating well with J_c values. The exponent n is conventionally used to characterize the sharpness of the transition, which is affected by various intrinsic and/or extrinsic factors. Taking into account the power law and Anderson's thermally stimulated flux creep model, the n-exponent can be identified with U_0/k_BT (where: U_0-averaged pinning potential, k_B-Boltzman's constant and T-temperature). The voltage rise is due to flux motion in the superconductor and is also influenced by factors leading to current sharing inside or among the non-uniform filaments. In our case, lower n-exponent for wire annealed at 600 ^{o}C can be understood by worse intrinsic filaments quality (see Fig. 2b). On the other side, lower contact resistance between the components in a composite (Fig. 2a) should reduce the effect current sharing inside or among the non-uniform filaments.

The temperature dependence of the critical current density, $J_c(T)$, of both wires at self-field is shown in Figure 3b. Similarly as for compared $J_c(B)$, the J_c values of 600 ^{o}C sample are lower due to lower T_c, see Fig. 2b. $J_c = 10^5$ Acm^{-2} is measured at 24.35 K and at 27.7 K for 600 ^{o}C and 800 ^{o}C annealing, respectively. On the other hand, the n values of 800 ºC sample were about 15-18 for T around 20-26 K, much lower than those measured for the 600 ºC wire, which are 50-60 for the same temperature range. This can be observed in the insert of figure 3b, where the I-V curves of both samples at self-field are displayed. One can see that n-values ratio at 4.2 K is contrary to those of high temperature measurement (20-26 K). The origin of different n-values at high temperature measurement in self-field (Fig. 3b) and low temperature measurement in applied field (Fig. 3a) is not fully understood. Possible reasons for explanation are: (i) higher current densities at temperatures 20-26 K and self-field can make the effect of current sharing on n-value more dominant that intrinsic filaments property, (ii)

pulse and direct transport current were used for high temperature (with cryocooler) and low temperature (at liquid He) measurements, respectively. Different current mode and cooling conditions can influence the estimated *n*-values. Moreover, the resistance per unit length of both wires at $T \approx T_c$ is very different: 0.19 mΩ/cm for 600 ºC and 1.6 mΩ/cm for 800 ºC wire, which correlates well with the results presented by Fig. 2 (0.21 and 1.63 mΩ/cm).

3.3. I-V curves

Figure 4a-b shows *I-V* curves of both wires measured at external field between 5.5 and 8 T. The dashed line marks the points of quenches, where the transport current is excluded from MgB_2 filaments and starts to flow only through the metallic elements. While the voltage measured at quench point (p_Q) is decreasing with increased transport current (decreased external field), conductor recovery takes place at nearly the same voltage level (≈ 0.01 V), see the dashed circle. Quench powers $P_Q = I_Q \times V_Q$ were evaluated and plotted for both wires using the date from Fig 4a and 4b. Figure 4c presents $P_Q(B)$ dependences which proves that the wire heat treated at 600 ºC is quenching at higher powers and is able to carry higher transport currents in stable regime. It can be attributed to its better stabilization due to less contaminated copper stabilization.

3.4. Normal zone propagation

For the normal zone propagation measurements, the length of the wire was 11 cm (9 cm between the current contacts and 1 cm for the contacts). In the experiments, the energy was deposited to the wire (carrying a current, $I < I_c$) by passing a rectangular current pulse of duration $t_p \sim 50$ ms) to a heater made of a graphite based paste (ECCOBOND 60L), which is placed in the middle of the wire. A typical measurement of the voltages obtained between adjacent voltage taps during a quench is plotted in Fig. 5, which corresponds to sample annealed at 600 ºC. The change of d*V*/d*t* at V_{23}, V_{34}, V_{45}= 5.6 mV reflects the change between the superconducting to the normal state.

Figure 6a shows the MQE and as function of the reduced current, I/I_c, obtained for both wires at different temperatures. Sample annealed at 600 ºC has higher MQE values than the wire annealed at 800 ºC. Therefore, for the point of view of the stability, the sample annealed at low temperature has better properties. The higher MQE values of sample annealed at 600 ºC are probably due to the lower resistivity of the sheath, compared with the wire annealed at 800 ºC, as mentioned previously. The MQE values depend on the resistivity of the metal matrix [16], so that by decreasing the resistivity of the wire increases the MQE values.

For example, for MgB_2 Cu-stabilized wires with a resistivity at T_c equal to: 1.6 mΩ/cm (for 800 ºC wire), 0.19 mΩ/cm (for 600 ºC wire) and 0.022 mΩ/cm (for wire analysed in [16]), MQE at I/I_c= 0.5 varies from 6 mJ to 10.5 mJ and 30 mJ, respectively. Therefore the amount of Cu in the wires as well as its resistivity, which depends also on the reaction of the Cu with the adjacent materials, are very important in view of the stability behaviour of superconducting/metal wires.

On the other hand, with regard to stability issues, high v_p values are always desirable, i.e. the rapid spread of the quench along the wire in order to avoid the local burning of the sample due to hot spots. v_p values are shown in Figure 6b as a function of I/I_c, for the same wires than in figure 6a and at the same conditions. v_p values have been calculated from the time delay between $V_{34}(t)$ and $V_{45}(t)$ curves, outside the zone between voltage taps 1 and 2, where the heater is placed, as it can be seen in Fig. 5. It is observed that sample annealed at 600 ºC has considerably higher v_p and therefore better properties that the wire annealed at 800 ºC. The higher slope of the v_p (I/I_c) curve can be explained by the higher n values of the I-V curve ($V \propto I_n$) of the sample annealed at 600 ºC (n = 50-60 at this temperature range), compared to the lower values (n = 15-18) for the wire annealed at 800 ºC.

The comparison between measured MQE and v_p values with numerical simulations have not been done for these conductors. Nevertheless, this has been done for other non-stabilized and Cu-stabilized metal/MgB_2 conductors [11, 16], and we found that a numerical model based on one-dimension heat diffusion with the measured finite n-values can satisfactorily describe the experimental observed quench behaviour of these conductors.

4. Conclusion

Multi-core Cu stabilized MgB_2 wire with Ti barrier and SS reinforcement annealed at 600 ^oC/2.5 h and 800 ^oC/0.5 h have been tested. Apparently increased wire's resistance was measured for the wire annealed at 800 ^oC/0.5 h. It is caused by more intensive reaction between the composite elements, especially between Cu and Ti. I-V curves measured far above the I_c level have shown that wire heat treated at 600 ºC is quenching at higher powers and is able to carry higher transport currents in stable regime. Minimum quench energy and normal zone propagation velocity are correlating well with wire's resistances and quenches measured by I-V curves. Consequently, the applied heat treatment conditions have to be considered carefully to maximize J_c but also to minimize the contamination of stabilizing copper.

Acknowledgement

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Figure captions

Figure 1. Cross-section of the wire observed by optical microscopy after HT at 600 ºC/2.5h.

Figure 2. $R(T)$ dependences of MgB_2/Ti/Cu/SS wires of 0.86 mm in diameter and 20 mm in length heat treated at 600 ºC/2.5 h and 800 ºC/0.5 h (a) and corresponding normalized resistive transitions (b).

Figure 3. $J_c(B)$ characteristics of compared wires measured at 4.2 K (a), the insert shows in-field n-exponents and $J_c(T)$ dependences measured above 20 K (b), the insert shows an example of I-V measurement at self-field for wire HT at 600°C/2.5h (T=24 K, I_c=54.5 A, n=60) and at 800°C/0.5h (T=26 K I_c=82.5 A, n=15). The length of the voltage taps was 3.6 cm.

Figure 4. I-V curves measured far above the I_c criterion at 4.2 K and external fields 5.5 – 8 T for wire HT at 600 ºC/2.5h (a) and for 800 ºC/0.5h (b). Quench powers P_Q calculated for both wires (c).

Figure 5. Measured voltages during a quench for sample annealed at 600ºC, at T=24 K and I=44 A=0.8I_c. From left to right V_{12}, V_{23}, V_{34} and V_{45} with voltage taps lengths of 15, 7, 7, 7 mm, respectively. The positions of the heater and voltage taps are schematically drawn in the figure.

Figure 6. MQE values for wires HT at 600 ºC/2.5h and for 800 ºC/0.5h measured between 22 and 26K at variable transport currents (a) and quench propagation velocities, v_p, as a function of current (I/I_c) for the same wires and conditions (b).

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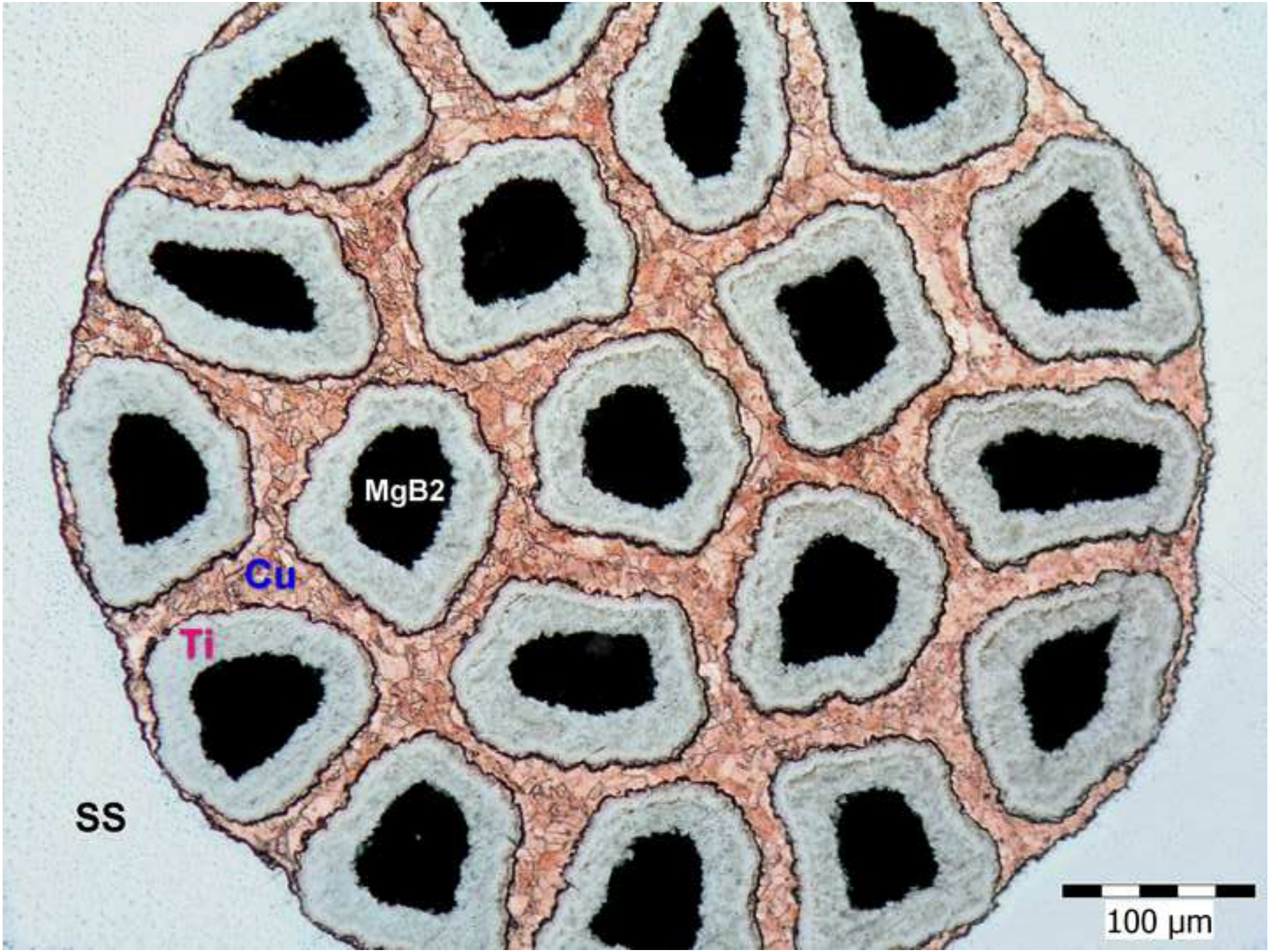

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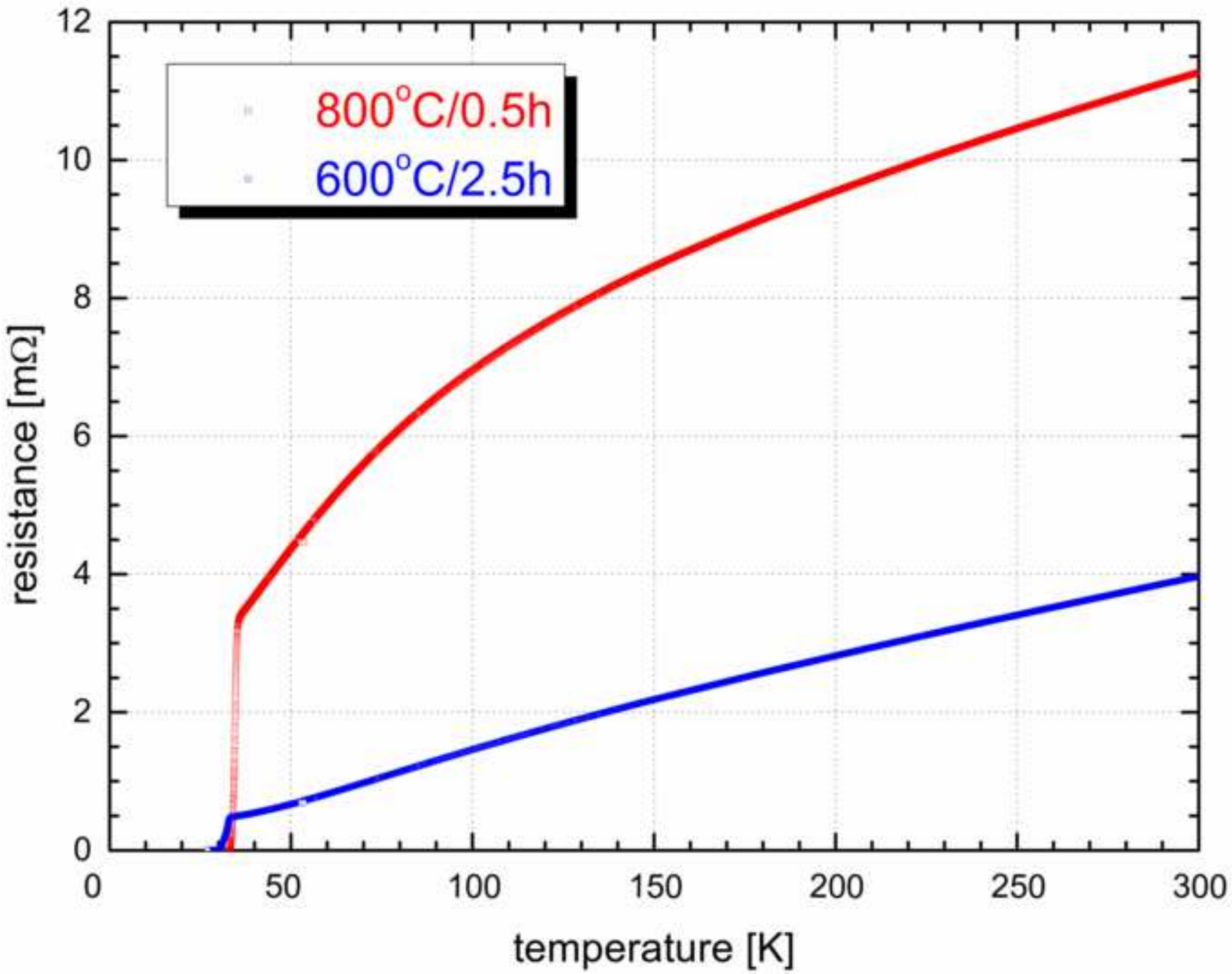

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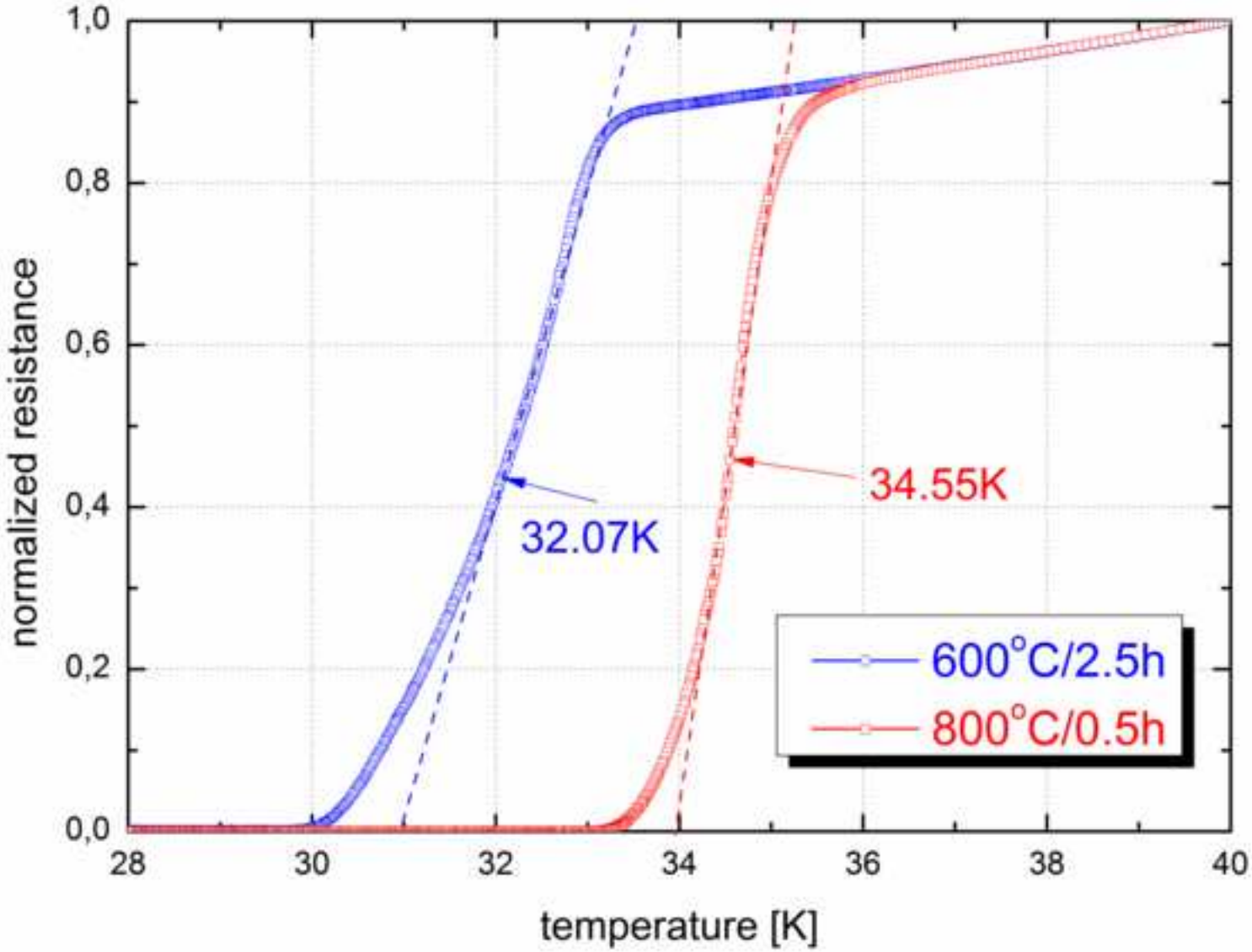

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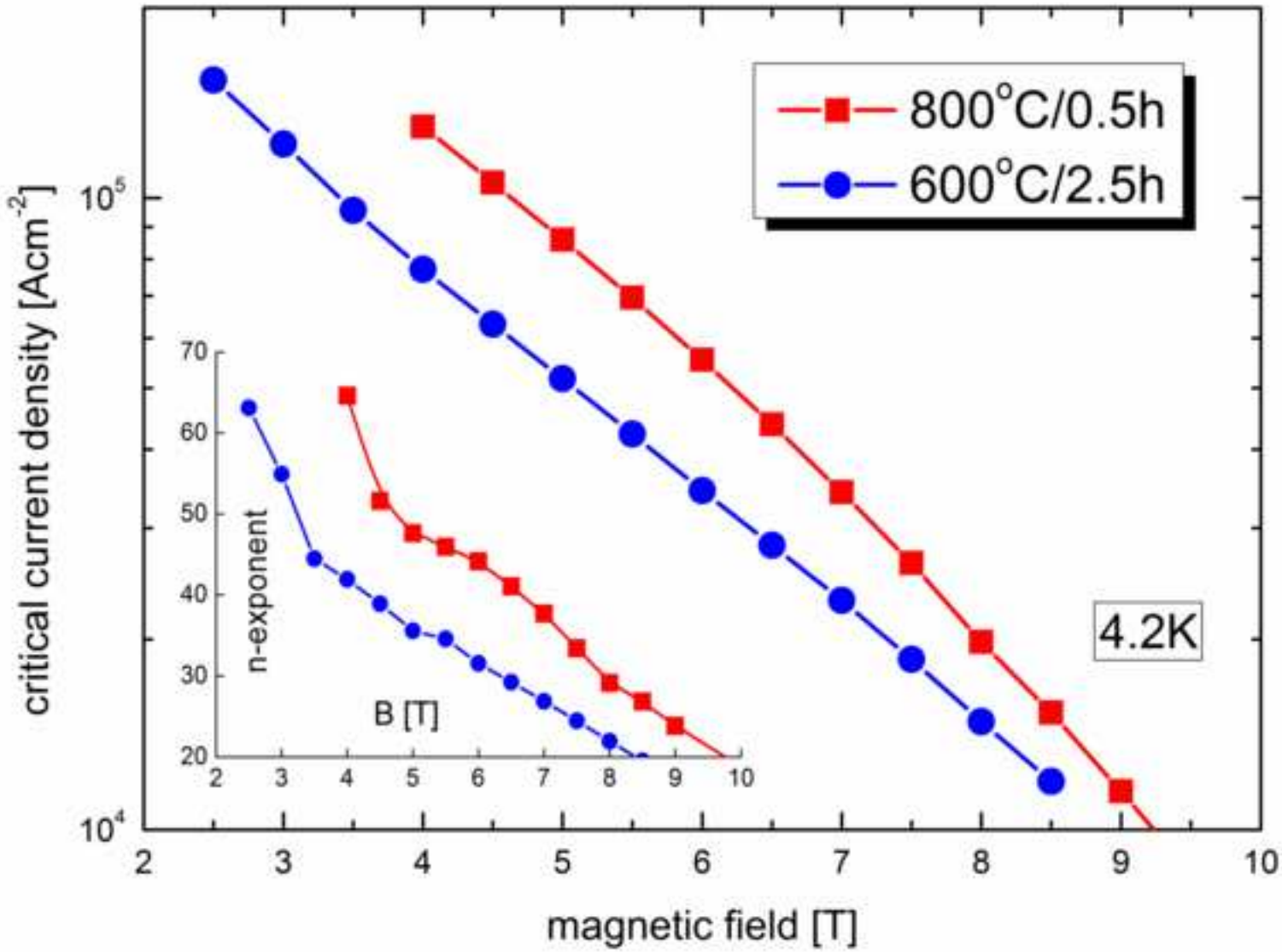

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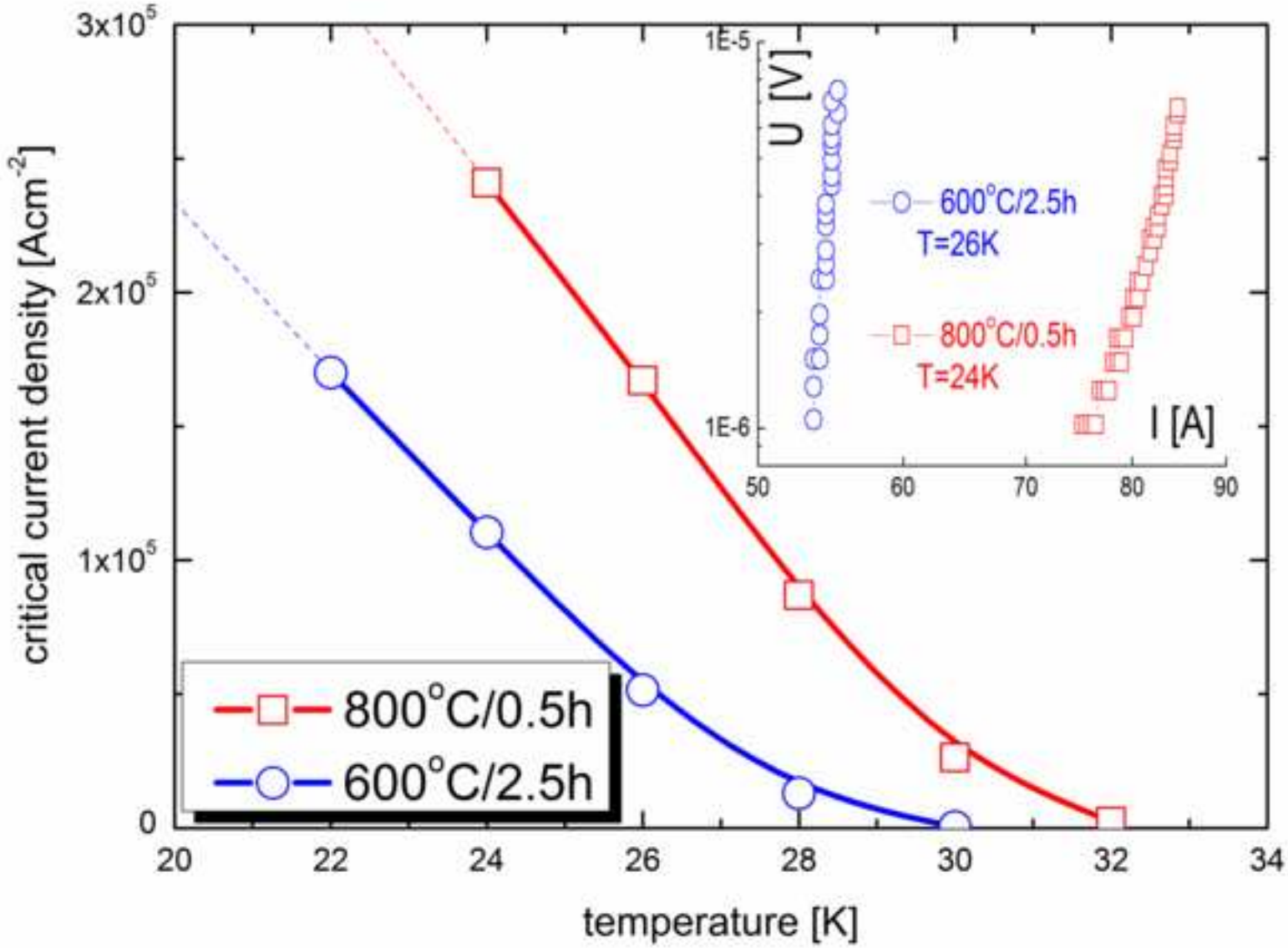

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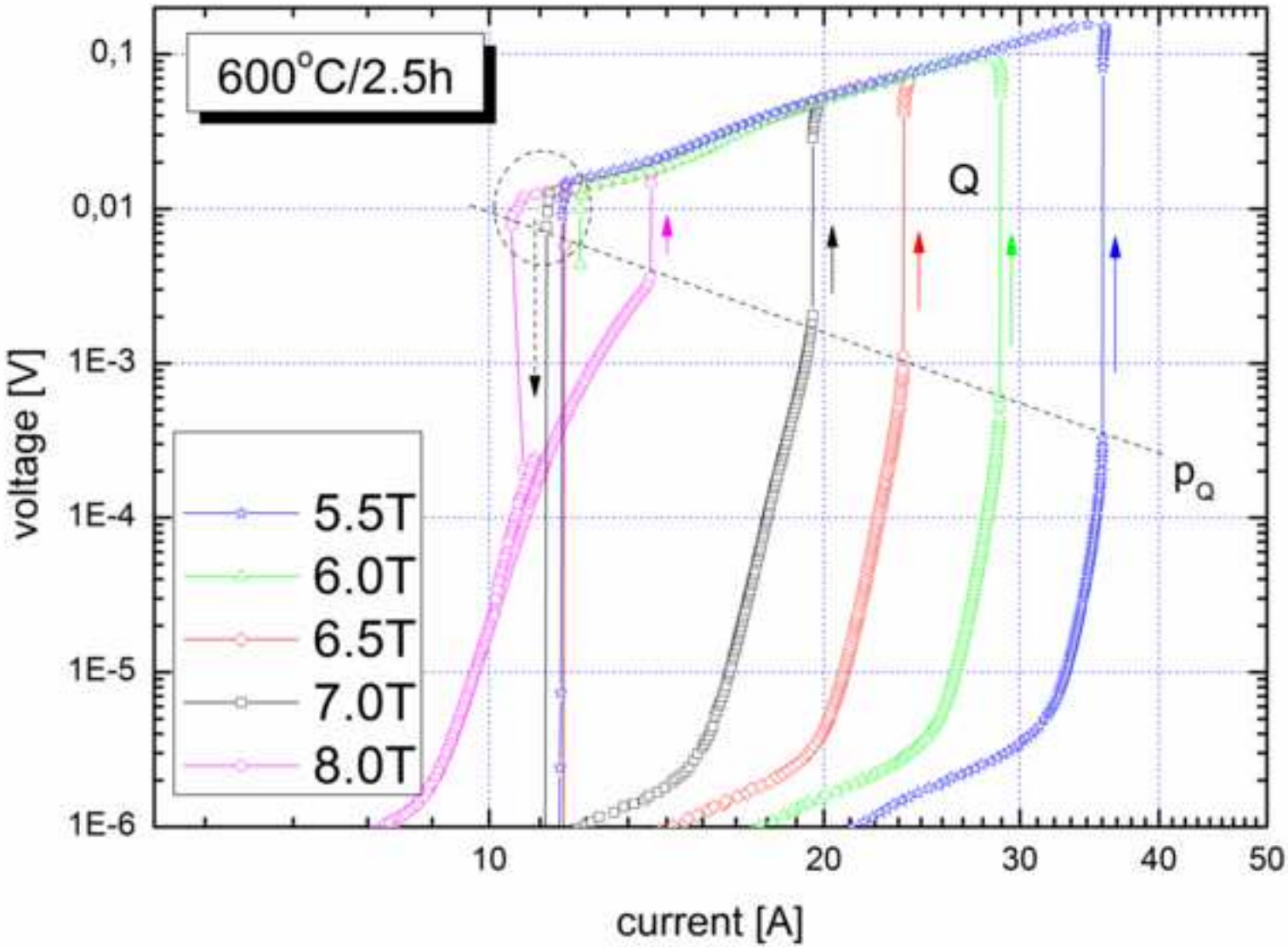

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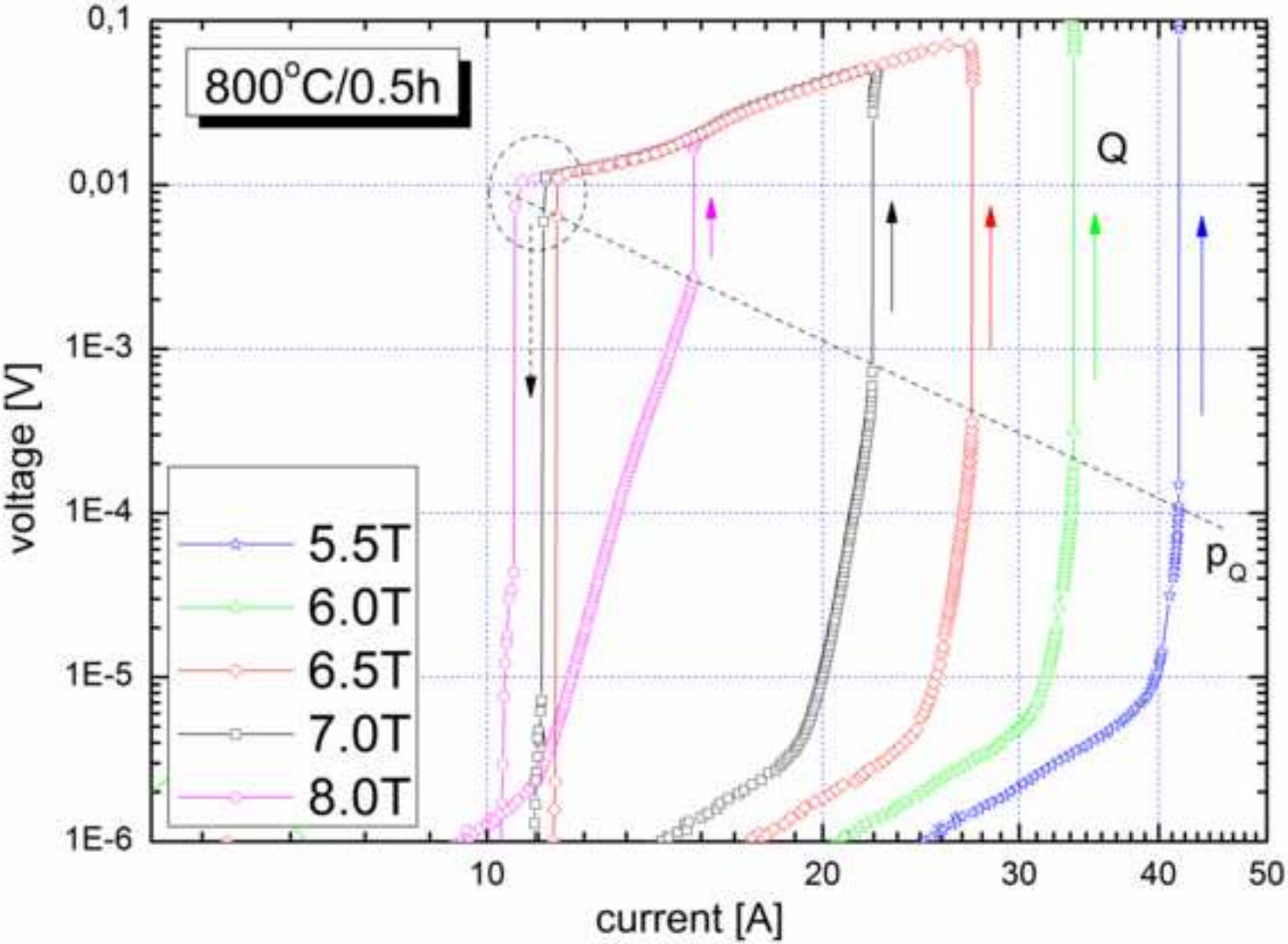

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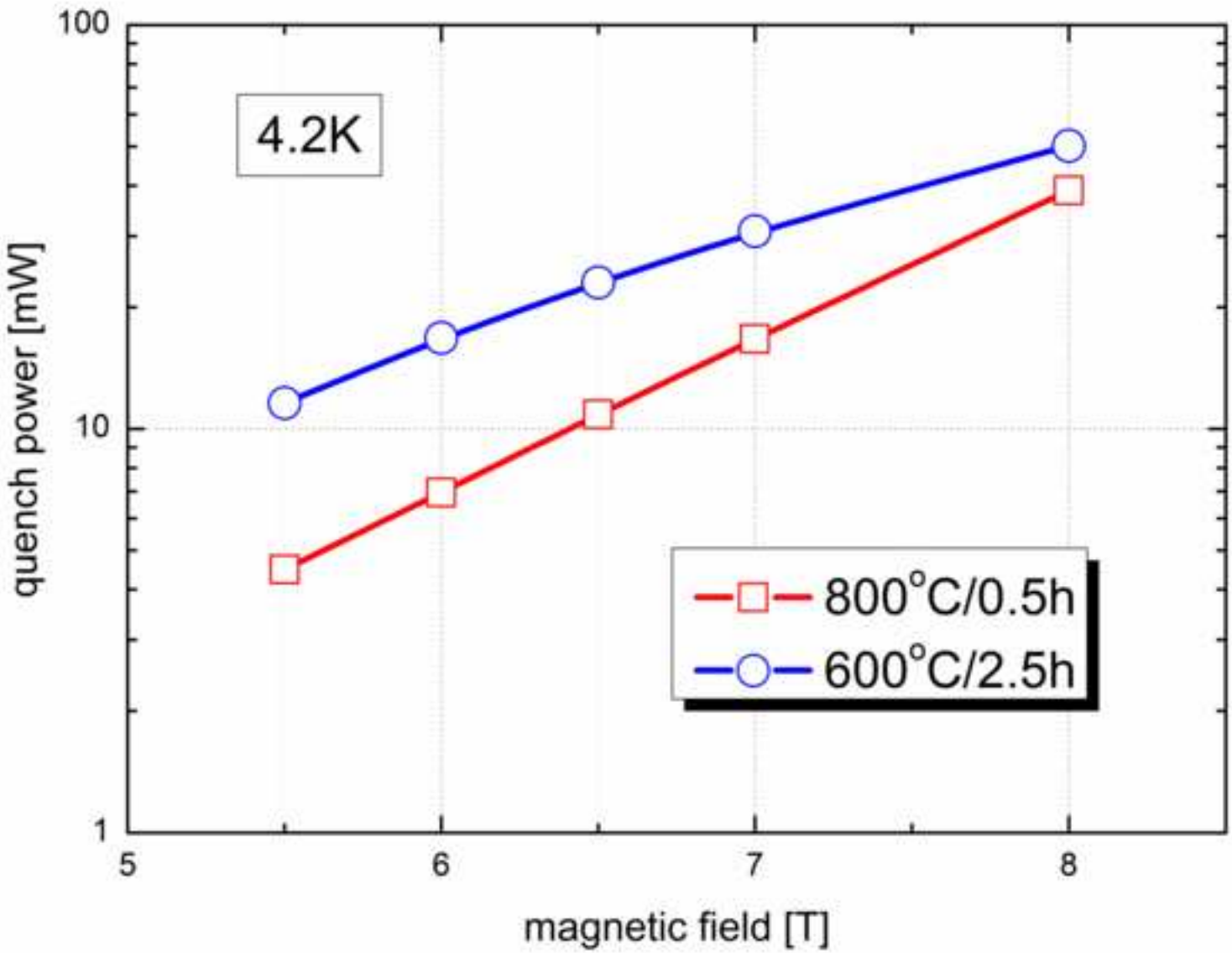

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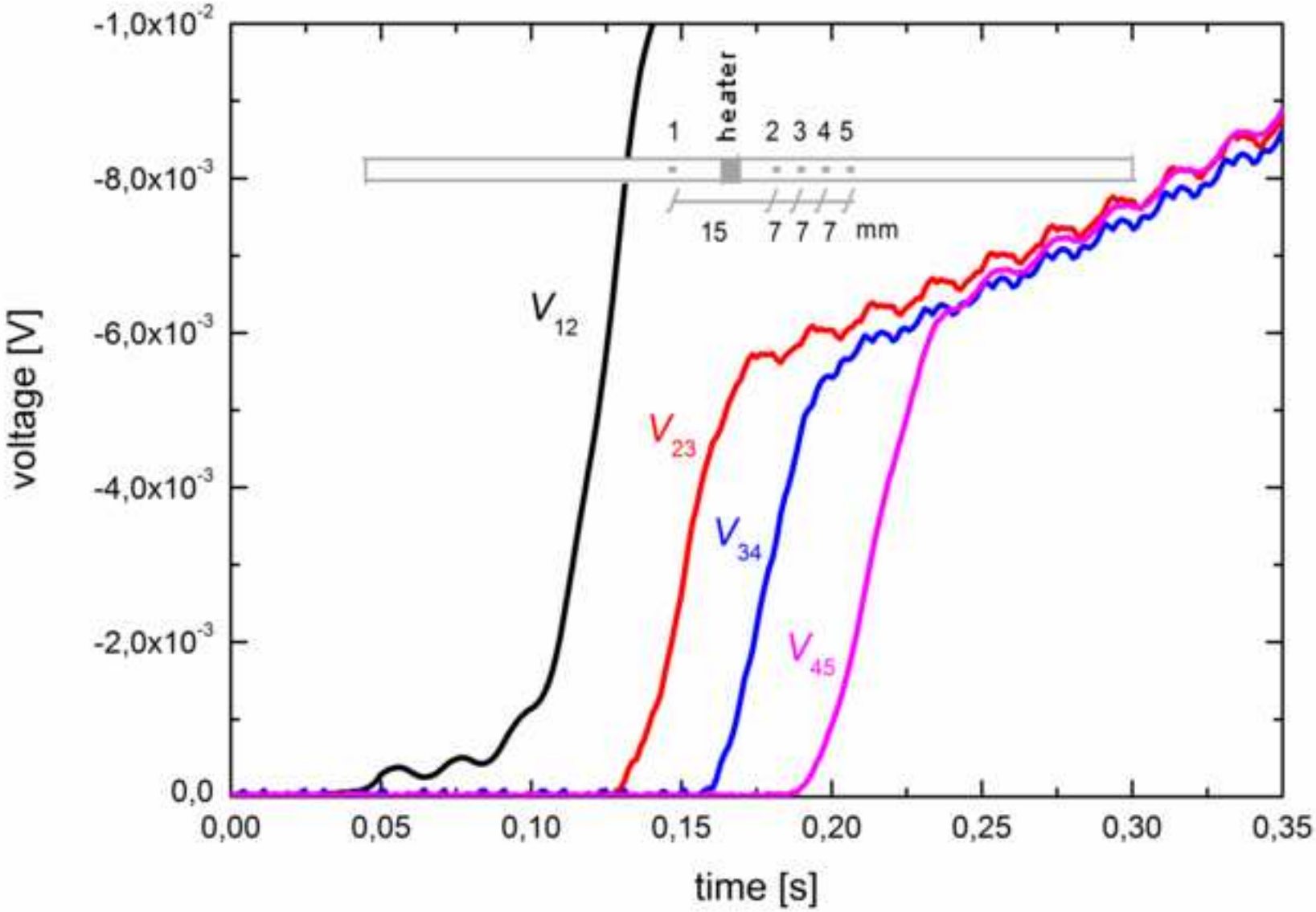

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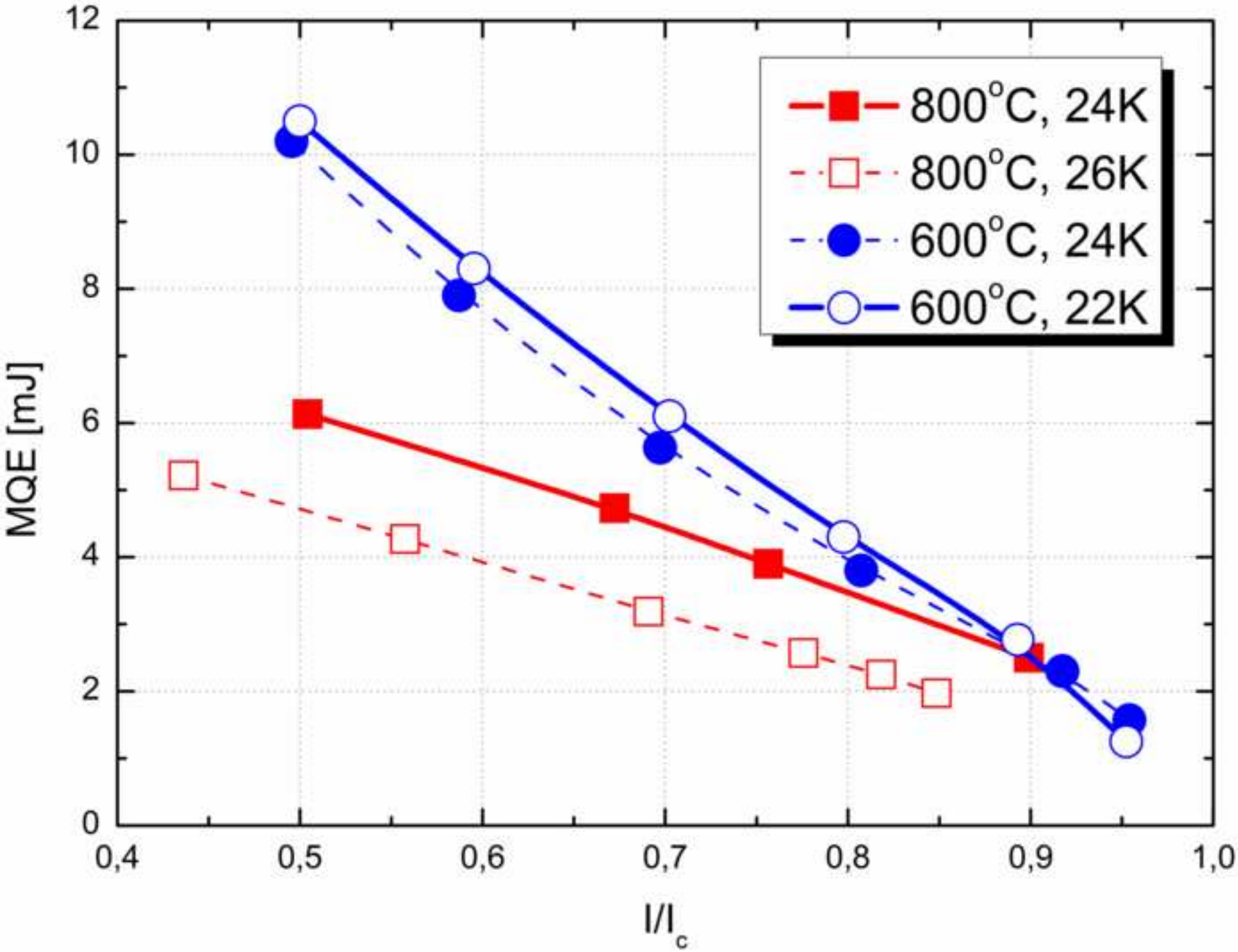

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