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A modified method to measure delamination strength of stabilizer free REBCO coated conductor under transverse tension

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A R T I C L E I N F O

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A B S T R A C T

A setup of transverse tension measurements of high temperature superconductor (HTS) RE-Ba-Cu-O coated conductors are modified for simplicity. Modification includes using high strength epoxy films instead of solder as adhesive agents and changing the shape of contact surface between test samples and anvils. This method was designed to measure delamination strength of stabilizer free REBCO coated conductors with a width more than 10 mm. It is also applicable to REBCO coated conductors with copper or stainless-steel stabilizers. Samples made by the IBAD/PLD process were tested by this test. Effect of operation temperature on the delamination strength of coated conductors was investigated. In addition, fracture surfaces of delaminated samples were studied.

1. Introduction

Second generation high temperature superconductor (HTS) RE-Ba-Cu-O (where RE represents a rare earth element) coated conductors (CCs) have potential to be used in high performance electrical equipment for their excellent electrical properties, including magnetic levitation systems and motors [1]. Fig. 1 shows a configuration of multilayer CC tapes, which consists of a substrate layer, buffer layers, a superconducting layer, a silver cap layer and a copper stabilizer layer. An extract lamination layer or an insulation layer may also be included for certain applications. CCs are commercially available these days. Annual yields are over a thousand kilometers worldwide according to the reports from manufacturers.

A typical manufacturing process of CCs can be divided into two stages in this study. The first stage is to produce "stabilizer free" "wide" CC tapes. Substrate layers (12 or 10 mm wide) are polished and go through multiple depositions to grow buffer layers, superconducting layers and silver cap layers. After an oxygenation process, these semi-finished tapes are characterized for quality control. All processing parameters are fixed in this stage for mass production. The 2nd stage is for tailoring final products specifically for different purposes. Parameters in this stage, such as the width of final tapes, the thickness of copper stabilizer layer, the choice of lamination layer, etc., are pre requested by end users, and therefore customized.

The multilayer structure of CCs leads to an anisotropy of mechanical properties. CCs with Hastelloy as substrates are known for their high strength in axial direction {fig1}, critical tensile stress of which can be as high as 700 MPa [2]. On the other hand, the adhesion between layers is much smaller. In high field applications such as insert magnet, MAGLEV and MRI, CCs coils are subject to Lorentz force in transverse direction [3]. Epoxy impregnation introduces thermal stress in cryogenic temperature [4]. The combination of Lorentz force and thermal stress have been calculated to be over 100 MPa [5], which may lead to a delamination of CCs and the failure of HTS magnet.

Mechanical behaviors of CCs have been studied comprehensively by using a delamination test. Mainly because research shows the critical current of CCs does not degrade significantly unless delamination initiated [6,7]. Studies of delamination behaviors of CCs under transverse tensile stress [8–10], peel stress [11], cleavage stress [12] and shear stress [7] have been reported. Most studies focus on customized CC samples after 2nd stage processing. Influence of stabilizer layers, width of CC samples, lamination and impregnation has been well studied for better coil preparation.

Our study mainly focuses on "stabilizer free" “wide” CC tapes, the product of 1st stage manufacturing. Therefore, finishes of CC tapes, including the width of tapes, cutting methods, stabilizer choices, etc., do not influence the test results. Instead, the methods to grow CC tapes and the processing parameters may have a more pronounced effect and be

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identified in the tests. We developed a simple, more general setup for delamination test of the fragile stabilizer free CCs. Hopefully, the effect of sample preparation, mounting etc., can be limited. This setup was applicable over a wide range of cryogenic temperatures so the influence of temperature on delamination strength was investigated. Finally, the mechanism of delamination under transverse stress was studied through microscope observation.

2. Details of delamination tests

Testing method in this study was based on transverse tensile tests. In this test, a test sample is sandwiched between a top anvil and a bottom anvil. Transverse tensile force is applied until the sample delaminates. The test setup was modified for stabilizer free CCs.

2.1. Replacing solder with epoxy

Solder such as Sn-Pb and In-Bi is often used to attach CC samples to top and bottom anvils. Unlike CCs surrounded by Cu stabilizer layers, the solder process of stabilizer free CCs is less straightforward because surface layers on top and bottom side of samples are different. On top of the superconducting layers is a silver cap layer, typically 1-2 micrometers. This requires a low temperature solder to prevent penetration of solder metal through the cap layer. On the opposite side is a substrate layer made of Ni-W, stainless steel or Hastelloy, leading to a different solder process depending on the choice of substrate materials [6].

To simplify the sample preparation process, epoxy adhesive instead of solder was used to attach CC samples to the anvils. This is because attachment of epoxy onto a selective surface may be less dependent on the surface materials. In this study, an assumption was made that the physical properties of adhesive agents have negligible influence on the result of delamination test, as long as samples can be firmly attached to test anvils. As a result, the only issue left was to find an epoxy strong enough at cryogenic temperature. A commercial adhesive film named FM-1000 was chosen in this study. It is made from modified polyamide and developed specifically for bonding metals. It claims to be serviceable over a temperature range from 20 K to 366 K. The cure temperature of this adhesive is relatively low, typically at 443±5 K, at which superconducting properties of CCs are not influenced.

2.2. Preparation of samples for delamination test

The handling of adhesive films is significantly easier. Here, the details of sample preparation are introduced. The modified procedures aim for better control of environment parameters and lowering possibility of failure sample. Hopefully, the variation introduced during the procedures can be minimized.

Fig. 2a shows a picture of a homemade fixture for quick sample assembly. The metal frame and pins can align the top and bottom anvils precisely, which enable the usage of identical anvils on both sides of the sample. The anvils are made by copper for better thermal conductivity. A platinum temperature sensor (PT100) is soldered to the bottom anvil as close to the sample as possible.

In a typical sample preparation, the adhesive films were first cut to the same shape of anvil cross-section by a paper hole punch, and then inserted between anvils and the sample. A spring was tightened to firmly lock the samples and anvils. The fixture and samples were put into a furnace for the cure of adhesives, which lasted for 4 hours at 463K. Virgin solid adhesive film liquefied and then solidified again to bond the HTS coated conductor with the anvils during a slow cooling to room temperature. 15 hours later, the mechanical strength of FM1000 adhesive film reached its maximum. The furnace used in this study has a uniform zone, allowing up to 4 sets of samples to cure together.

The anvils can be machined to different shapes for either round or rectangle contact sections between samples and anvils. A rectangle contact section is applicable for almost any type of CC samples by simply adjusting the aspect ratio, while a round contact section requires a much wider sample than the contact area. On the other hand, a round contact section avoids sharp edges and corners. Fig. 3 shows a COMSOL analysis comparing the influence of anvil shapes. The stress distributes more homogeneously in the case of round contact section, which may improve the reliability of test data. In this study, round contact sections of 4 mm diameter were used for the uncut samples (10 mm or 12 mm in width).

2.3. Procedure of delamination test

A sample, as shown in Fig. 2c, was connected to a universal mechanical testing machine through two pairs of pulling rods. Pulling rods were linked by quick release locking pins following a perpendicular rule, as shown in Fig. 4a. This setup allows free rotation of pulling rods around pins in both X and Y direction, which minimizes the shear force on the sample. The transverse load was applied to the sample at a displacement rate of 0.5 mm/min until actual delamination.

The above test can also operate in a liquid nitrogen bath. A sample was fully submerged in a tank of liquid nitrogen for 15 minutes before application of transverse load. The tank can be elevated by an electric motor for installation and removal of samples, as shown in Fig. 4b.

Moreover, the test can be performed at any temperature between boiled point of liquid nitrogen and ambient condition. Instead of being submerged in liquid nitrogen, a sample was hung above the surface of liquid nitrogen. The sample was cooled by conduction of bottom anvil and convection of boiled nitrogen. Temperature of the sample was monitored by a PT100 temperature sensor until a stable temperature was reached. This temperature was recorded as reference temperature. The transverse load was then applied to perform the delamination test. The temperature can be adjusted roughly by moving the liquid nitrogen tank upward or downward. Unlike in a liquid nitrogen bath, the sample was not in a thermal equilibrium condition. A temperature gradient along the z direction must be considered carefully. In the case of very thin cc tapes, the temperature difference of top and bottom surface

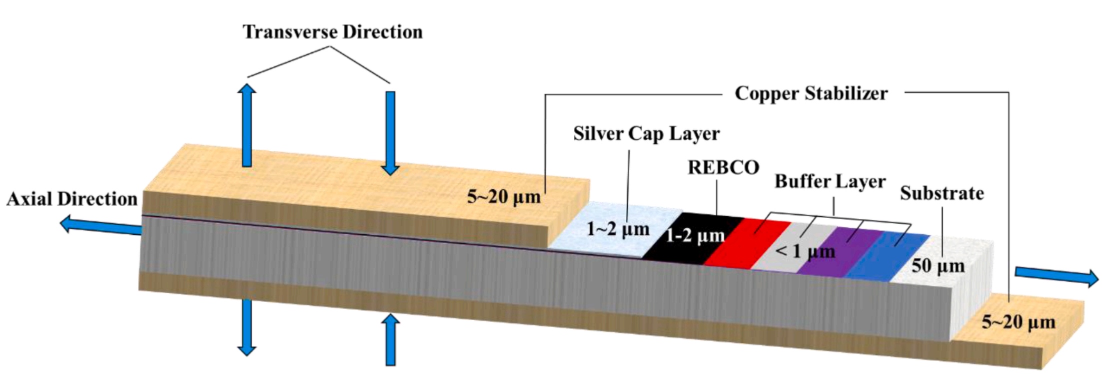

Fig. 1. schematic drawing of a CC tape

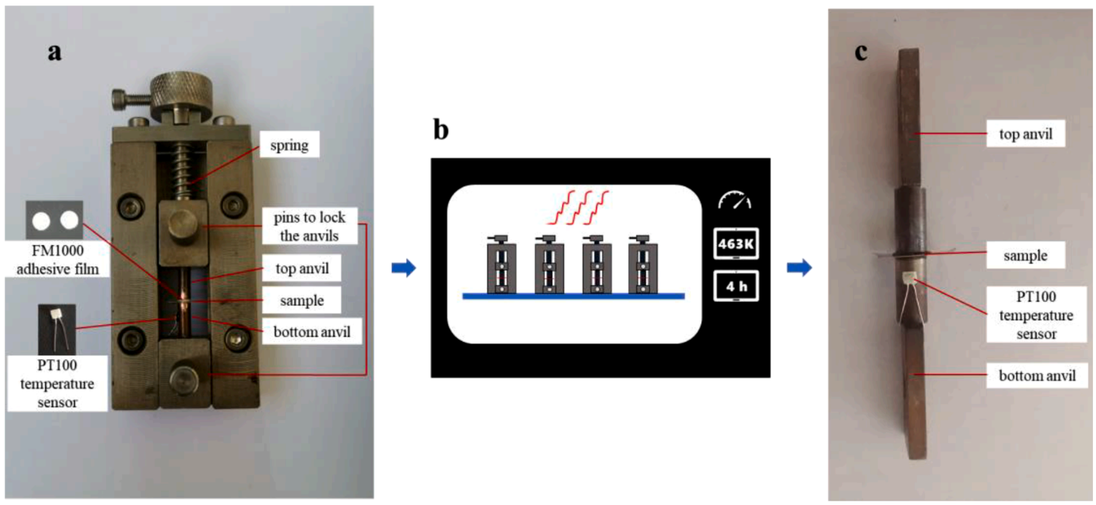

Fig. 2. a) fixture used for sample preparation b) cure of adhesive c) samples ready for delamination test

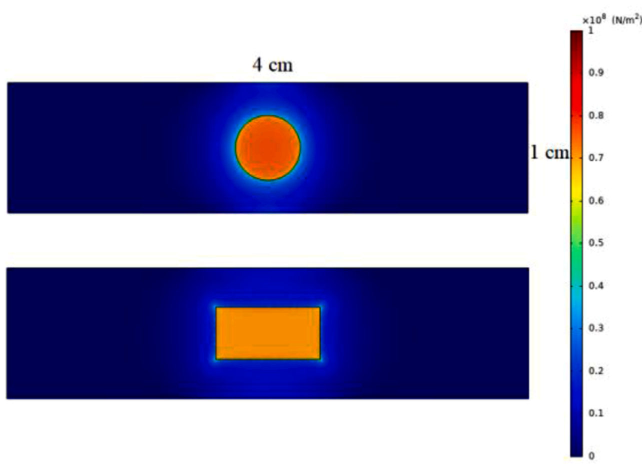

Fig. 3. COMSOL analysis results for the stress distribution on the surface of REBCO coated conductor tape formed by cylindrical anvils and square anvil under the same tensile force.

should be neglectable.

2.4. Samples

Details of samples tested in this study are listed in Table 1. Samples named after A or B were cut from a 4-meter homemade CC tape in the same batch of IBAD/PLD process. Each sample was at least 20 mm long. An extra copper stabilizer layer was electroplated on sample Bs. Samples named after D, E and F were chosen from tapes made in different batches with adjusted process parameters. Samples cut from a commercial CC tape were used as reference, named after C.

3. Experiment results and discussion

3.1. Verification of the modified method

The transvers behaviors of CCs are considered non-uniformly distributed, reflecting both the variation in test setup and the intrinsic material properties such as defect geometries and structures. A qualified test setup should minimize the former effect and correctly reflecting the intrinsic properties of test samples. To verify the proposed method, 3 sets of samples were selected for comparison. Difference of sample parameters were operation temperature, manufacturing process, batch and batch difference.

3.1.1. 77K vs room temperature

Delamination strength of sample A and B were measured at liquid nitrogen and room temperature (RT), with a sample size of 188 (shown in Fig. 5a).

At RT, delamination strength of sample A has a value of 46.98 MPa at minimum and 88.91MPa, at maximum, averaging 70.48 MPa. Delamination strength of sample B ranges from 29.75 MPa to 85.04 MPa, with an average value of 63.65 MPa. At 77 K, the maximum and minimum measure results of sample A is 164.97 MPa and 82.08 MPa, averaging 126.57 MPa. At the same temperature, test results of sample B range from 96.17 MPa to 157.48 MPa with an average value of 129.52 MPa.

The measure results of sample A and sample B at the same temperature are very closed. It seems that a thin electroplated copper stabilizer does not affect the delamination strength at all.

The testing method is sensitive enough to reflect the impact of liquid nitrogen bath, as the measure result almost doubled at 77 K. The difference is more pronounced than the pin pull study reported in 2007 [6]. It is worth noting that delamination never happens below 80 MPa among the 77K tests. This may indication a high mechanical tolerance for application of CCs in liquid nitrogen.

3.1.2. Samples made by different manufacturing process

A commercial tape (sample C) made by a different fabrication process was used as the reference sample. Fig. 5c shows a similar delamination pattern at 77 K and RT. At 77 K, the measurement value of sample C is between 85.67 MPa to 155.02 MPa, and the average value is 115.15 MPa. At RT, the maximum and minimum measure results of sample C are 76.04 MPa and 41.88 MPa, and the average value is 59.05 MPa.

Compared to the test result shown in Fig. 5a, sample C has a slightly smaller delamination strength compared to sample A and B. On the other hand, the increase of delamination strength CCs at 77 K is not limited to samples made by the PLD process.

3.1.3. Samples made in different batch of production

Samples (D, E and F) selected from three batches of REBCO coated conductor tapes were also study in the form of black box testing. Processing parameters were adjusted between those samples. As shown in Fig. 5d, average delamination strength of sample D, E and F are 43.06 MPa, 57.33 MPa and 47.04 MPa. Sample E shows the highest max/min/avg values in the delamination test. On the other hand, the results for sample A and F are indistinguishable.

The parameter difference was revealed after the delamination test.

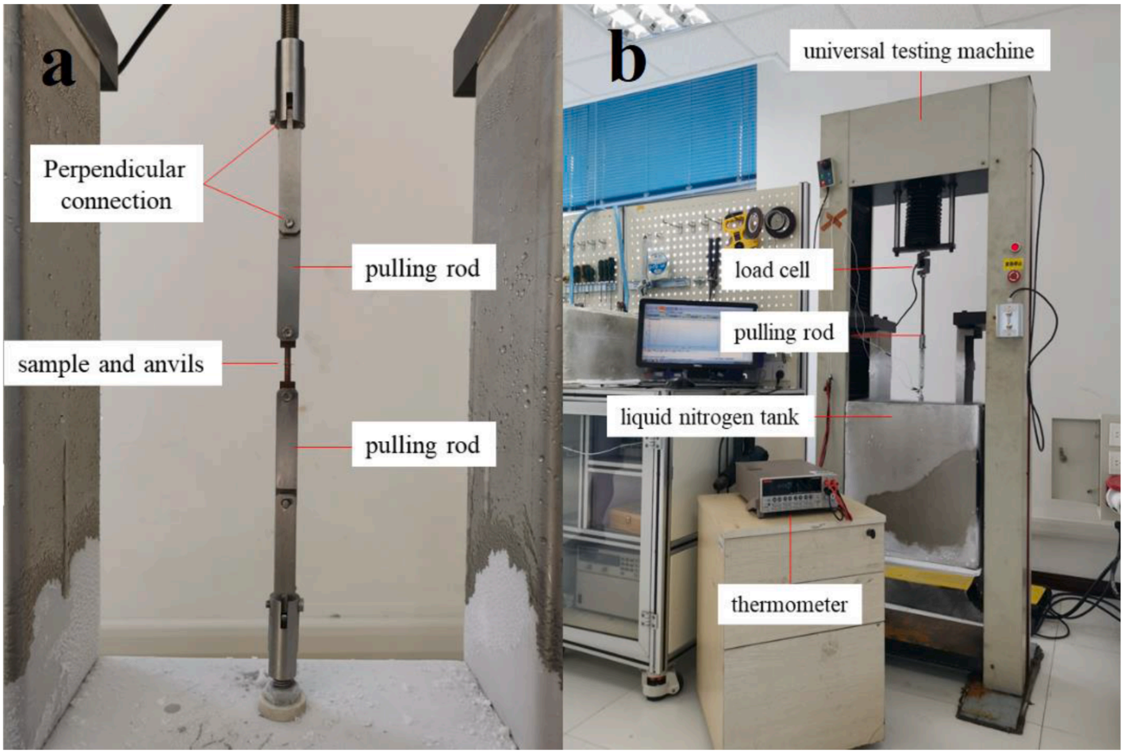

Fig. 4. (a) Installation of samples for delamination test (b) Overall view of the test setup

Table 1
List of Samples

	Sample A	Sample B	Sample C	Sample D E F
Fabrication process	IBAD/PLD	IBAD/PLD	IBAD/RCE-DR	IBAD/PLD
Substrate	Hastelloy	Hastelloy	Stainless steel	Hastelloy
Ic, A	>270	>270	>650	>270
Width, mm	10	10	12	10
Stabilizer	none	Electroplated copper (20μm)	none	none

Processing temperature of superconducting layer increased from sample D to E and F. Critical currents of all three samples are indistinguishable. This indicate the transvers test could be used as a complementary test alongside critical current measurement.

3.2. *Influence of operation temperature*

To further investigate the influence of operation temperature on the delamination strength of CC tapes, samples A and B were tested at temperature between 77 K and RT, as shown in Fig. 5b. Only one sample can be tested in each temperature due to the nature of the cooling method in this study. A clear trend that delamination strength increases as temperature drops can be observed, despite a relatively small sample size. This may indicate a further improving delamination strength at even lower temperature. Straight lines of best fit of data were drawn to estimate the delamination strength of CC tapes at cryogenic temperature. For example, delamination strength of cc tapes may be as high as 160 MPa at 4.2 K, as suggested by the line fitting. The equation of linear fitting of sample A is as follows:

$$y = -0.33795x + 165.83975$$

3.3. *Investigation of fracture samples*

Pictures of typical samples after the delamination test are shown in Fig. 6a and b. Silver cap-layers were completely removed after delamination (Fig. 6a) for 87.6% of all test samples, despite the test temperature. This proves that the FM1000 film has good adhesiveness on silver and copper surfaces.

An exposed buffer layer can be observed in 76.6% of all delaminated samples. The boundaries between exposed buffer layers and remaining superconducting layers have great contrast in optical microscopes. Fig. 6c and d are optical images of a delaminated sample A and C. Fractures of superconducting layers may happen somewhere at the boundaries, causing the removal of superconducting layers above the exposed buffer layers. Irregular structures were found at the boundaries, as shown in both figures. The composition of those region was consistence with the superconducting layer. It could be large area (~100 μm) of non-textured superconducting layer. The evolution of boundaries was interrupted by those structures.

4. Conclusion

To study the delamination strength of stabilizer free CC tapes, a modified transverse tensile testing method was developed. Usage of epoxy adhesive has greatly simplified the sample preparation process, which is ideal for mass testing. Reliability and sensitivity of the testing method was verified in this study.

Temperature dependence of delamination strength of CCs was investigated at temperature between 77 K and room temperature. Decreasing operation temperature can significantly improve the delamination strength of CCs. At 77 K, the critical stress is almost double compared to the result at RT. According to our estimation, the delamination strength can reach as high as 160 MPa at 4.2 K.

Declaration of Competing Interest

We declare that we have no financial and personal relationships with

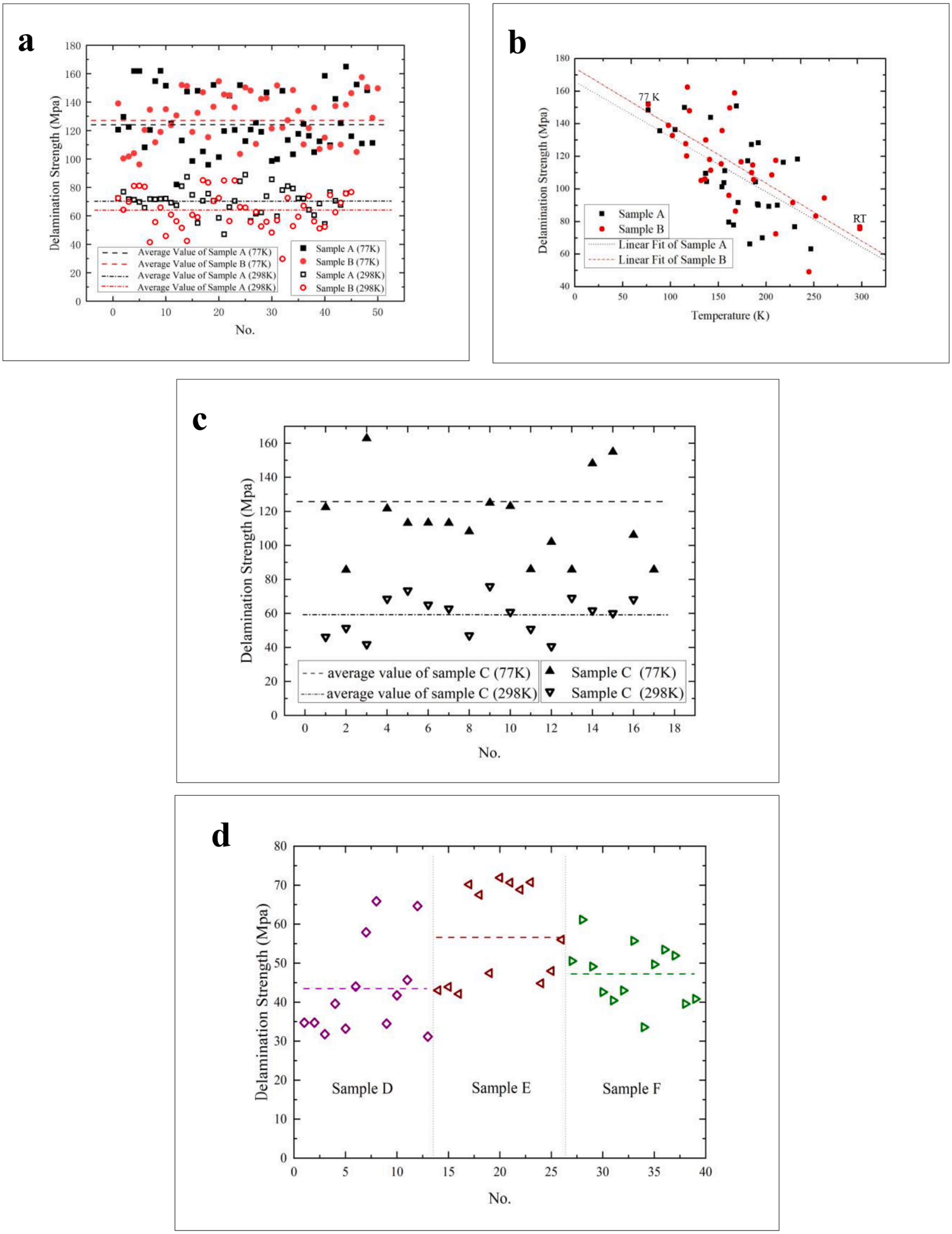

Fig. 5. (a) Delamination strength of sample A and sample B at 77K and Room temperature (RT). (b) Delamination strength of sample A and sample B at temperature range between 77K to RT. (c) Delamination strength of sample C at 77K and RT. (d) Delamination strength of sample D, E and F at RT

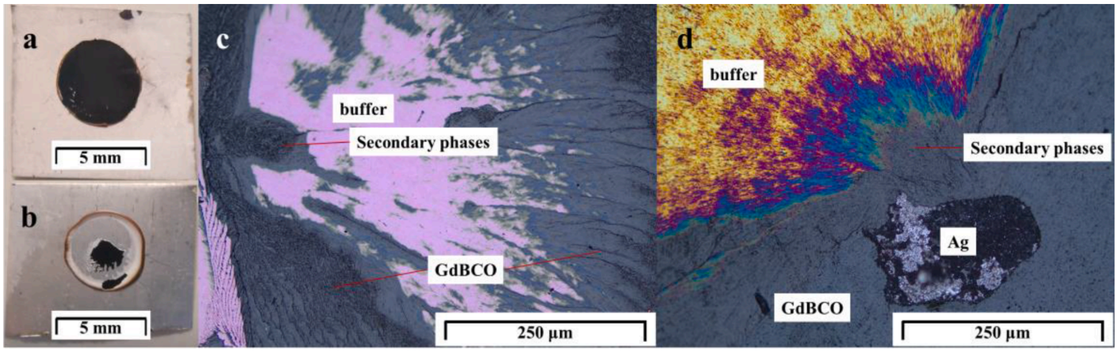

Fig. 6. (a) a sample delaminated from the edge of anvils (b) a sample delaminated from central part of anvils (c) a microscopic image of fracture area in sample A (d) a microscopic image of fracture area in sample C

other people or organizations that can inappropriately influence our work, there is no professional or other personal interest of any nature or kind in any product, service and/or company that could be construed as influencing the position presented in, or the review of, the manuscript entitled.

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