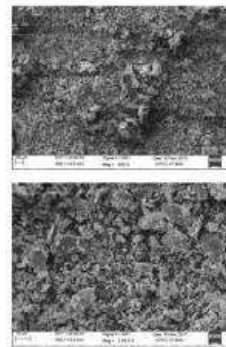
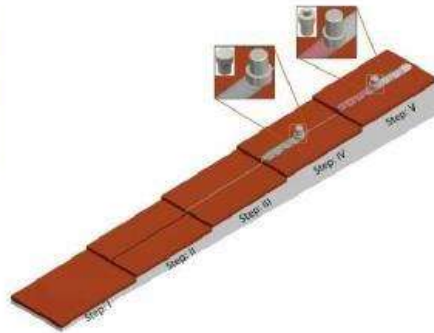


Chapter 3

Materials and Methods

Matrix and particulates



Fabrication of SCs

This chapter describes the details of materials used commercially pure copper and reinforcements (FA, Zircon sand and zirconia) for the present investigation. The experimental details involved for surface composite fabrication has been detailed thoroughly.

The chapter concludes with the details of the characterization techniques involved in the present investigation.

3.1 Introduction

This chapter describes the materials (Matrix and particulates) used for the present investigation and experimental details of the processing technique which has been used for the manufacturing of surface composites. The details of the tests followed in the present investigation such as mechanical characterization, tribological characterization, leaching test and electrical characterization has also been given in this chapter. Characterization such as optical microscopy, scanning electron microscopy and electron back scattered diffraction and X-ray diffraction are described.

3.2 Materials

In the present investigation, commercially pure copper has been used as matrix. Copper was procured from Tremor Alloys, Mumbai in the form of plates of dimensions 2000 mm x 100 mm x 6 mm which was further machined on band Saw in the dimensions of 150 mm x 100 mm 6 mm for final experimentation. The optical micrograph and chemical composition of commercially pure copper are shown in Fig. 3.1 and table 3.1 respectively.



Figure 3. 1 Optical micrograph of commercially pure copper

Table 3. 1 Chemical composition of commercially pure copper

Elements	P	As	O	Sb	Te	Cu
wt.%	0.0003	0.0005	0.0005	0.0004	0.0002	Balance

The grain structure of commercially pure copper is coarse with lots of twins. Three different particulates have been used as reinforcements for the fabrication of copper-based surface composites. Two of them i.e. fly ash and zircon sand which is considered as wastes were obtained from Hindalco industry, Renukot and a company of Orissa respectively. The third particulate (zirconia) was procured from Gyan scientific agencies, Varanasi. The chemical composition of zircon sand and fly ash has been shown in Table 3.2 and Table 3.3 respectively.

Table 3. 2 Chemical composition of Zircon Sand

Elements	ZrO₂	SiO₂	Al₂O₃	TiO₂	Fe₂O₃	SiO₂ (Free)
wt.%	65.90	32.54	1.15	0.27	0.04	0.10

Table 3. 3 Chemical composition of FA

Elements	SiO₂	Al₂O₃	Fe₂O₃	K₂O	TiO₂	CaO	P₂O₅	MgO	SO₃	Na₂O
wt.%	52.53	30.01	7.53	1.98	1.79	1.15	0.45	0.32	0.02	0.02

The SEM micrograph of the three particulates and their corresponding particle size distribution have been shown in Fig. 3.2.

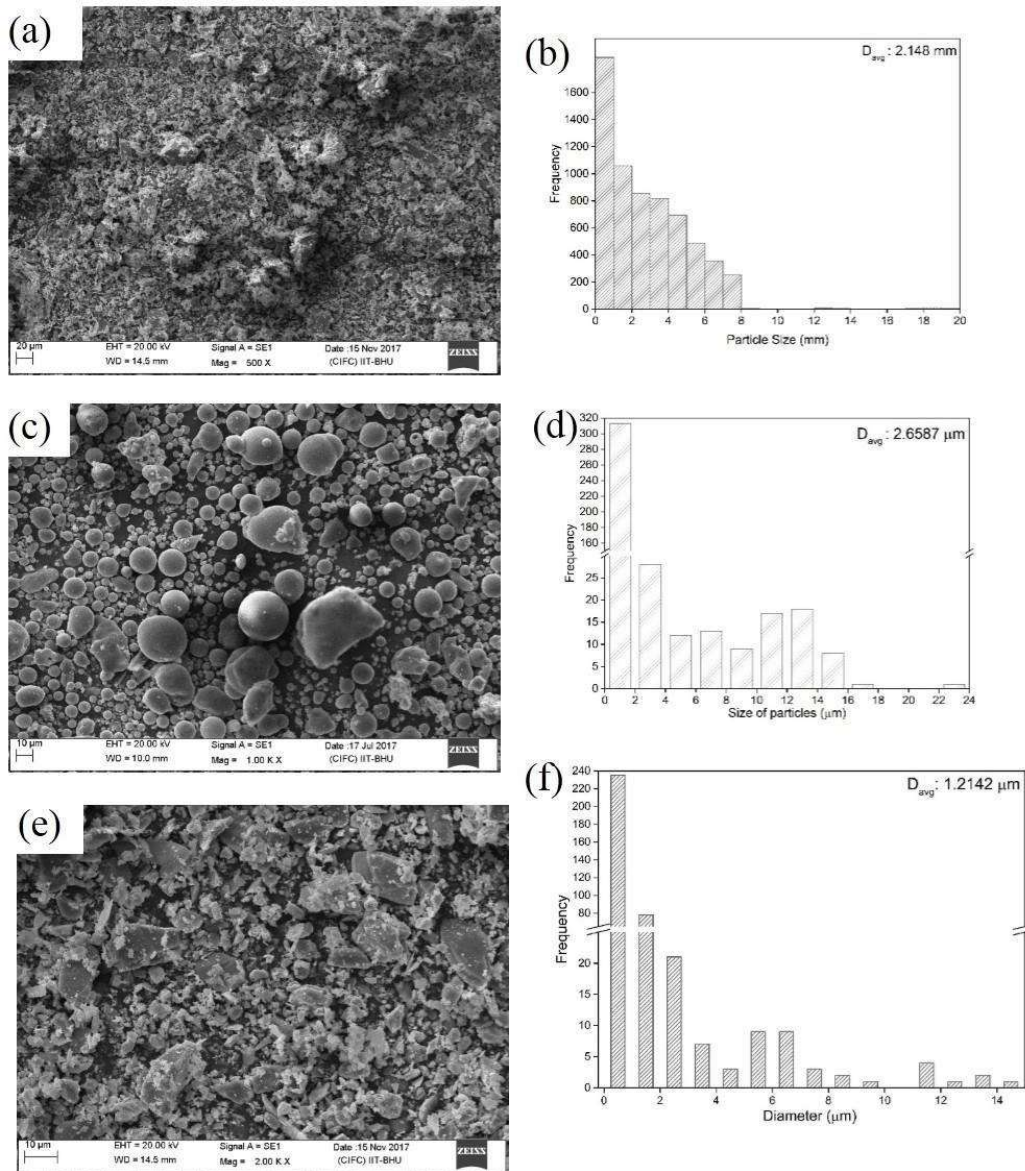


Figure 3. 2 SEM micrograph of the particulates and particle size distribution (a) and (b) zircon sand, (c) and (d) fly ash and (e) and (f) zirconia

The estimated average grain size for the particulates fly ash, zircon sand and zirconia were 2.65 μm , 2.14 μm and 1.21 μm respectively. The zircon sand obtained from the industry was of bigger grain size as shown in Fig. 2.3.

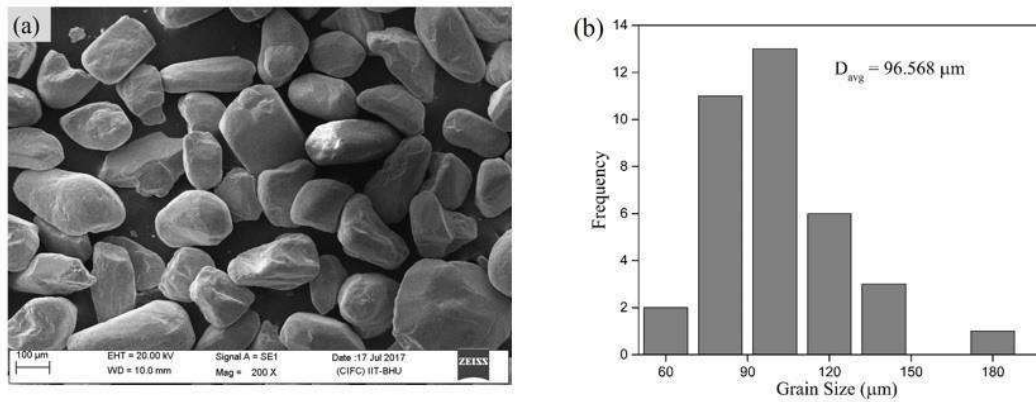


Figure 3.3 (a) SEM micrograph of as received zircon sand (b) particle size distribution

It can be observed that the particles are of bigger size having uneven surfaces with lots of sharp edges. The received zircon sand was ball milled to reduce the average grain size. The estimated average grain size of the receive zircon sand was 96.56 μm. The reduced average grain size of zircon sand with their particle size distribution is shown in Fig. 3.2.

3.3 Surface composite fabrication by FSP

The set-up used for composites fabrication (FSW Machine) is shown in Fig. 3.4.



Figure 3. 4 Friction stir processing set-up

The specially designed NC controlled friction stir welding machine used for present investigation was supplied by R. V. Machine Tools Coimbatore.

Sequential steps that involved in the fabrication of surface composites by FSP were machining a groove exactly at the centre of the work-piece viz. copper plate by CNC end mill cutter. Groove dimensions are usually cut according to the Vol% of reinforcements that has to be compacted within the slot. The volume fraction of reinforcements was calculated as (Tjong and Ma, 2000):

$$\text{Volume fraction} = \frac{\text{Area of Groove}}{\text{Projected area of tool pin}} \times 100$$

$$\text{Area of groove} = \text{Groove width} \times \text{Groove depth}$$

$$\text{The projected area of tool pin} = \text{Pin diameter} \times \text{Pin length}$$

As in the present investigation, the volume fraction of the reinforcements (Fly ash, zircon sand and zirconia) had to be 18 Vol%, so accordingly groove dimension was selected. The dimensions of the grooves were 1.5 mm in width and 3.5 mm in depth. In the next continuous step, the particles were carefully filled in the slots created over copper plates. Then compaction of particulates into the slots was carried over followed by passing of a pinless tool made of double tempered H13 tool steel. This procedure was performed to avoid the scattering of particles anywhere outside the groove during the stirring process. The final step of processing involves passing of FSP tool made of double tempered H13 tool steel above the copper surface with a 6 mm diameter pin of length 4 mm possessing a shoulder diameter of 18 mm. Pin profile was cylindrical and it was threaded. For comparison purpose, pure copper was FSPed with same processing parameters as used for composites. The FSPed plate without reinforcement were characterized under same conditions as for the composites.

Fabrication of surface composites was carried out on an NC friction stir welding machine. Fabrication was done under normal conditions with constant processing parameters which were decided on the basis of a literature survey and vigorous trial runs. The processing parameters used for these experiments were as follows: tool rotation speed (1000 RPM), traverse speed (40 mm/min) and tool tilt angle with the vertical (2.5°). The sequence of steps followed is as that shown in Fig. 3.5 for easy reference.

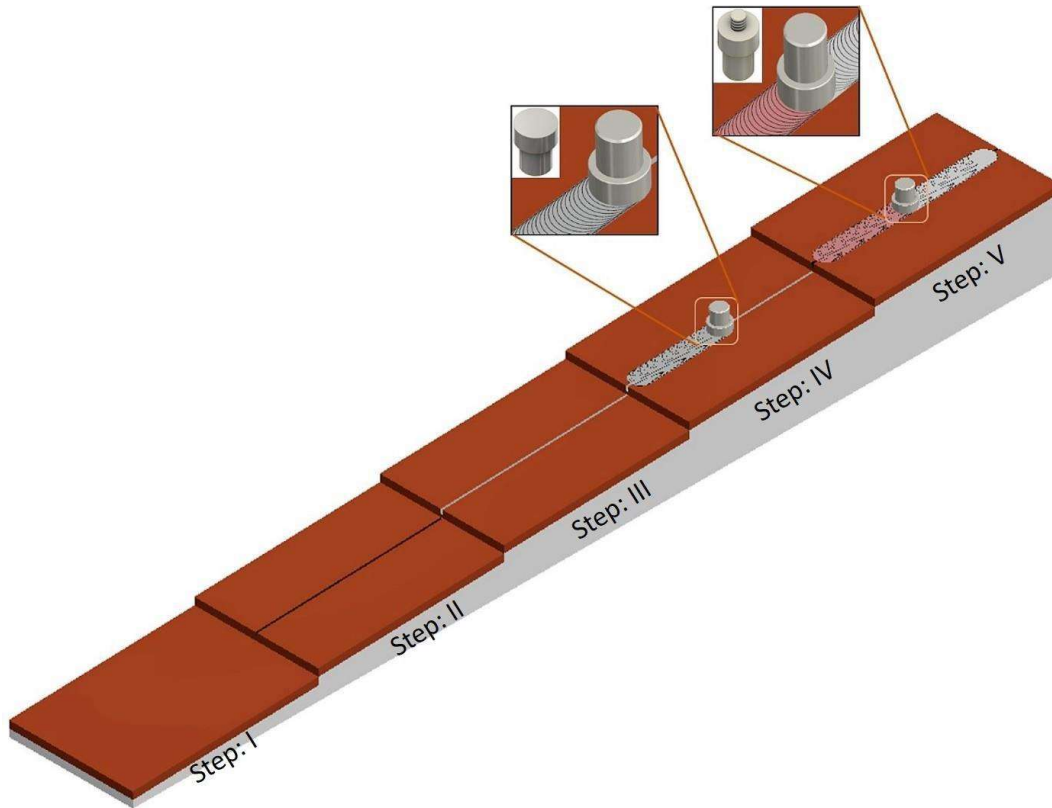


Figure 3. 5 Schematic views of FSP process in steps: I Plate of known dimension
 II Groove machining on the copper plate III compacting particulates in the
 grooves IV Groove closure by passing pinless tool V Friction stir processing

3.4 Digital Photography

The base commercially pure copper and processed copper with and without reinforcements samples were visually examined at every stage of experimentation to have a better understanding of the surface features such as depressions, irregularities and ring like pattern by using a digital camera.

3.5 Characterizations

Specimens for various characterizations were machined from base copper and FSPed copper with and without reinforcements. The strategies followed for samples extraction for various characterizations have been shown schematically in Fig. 3.6.

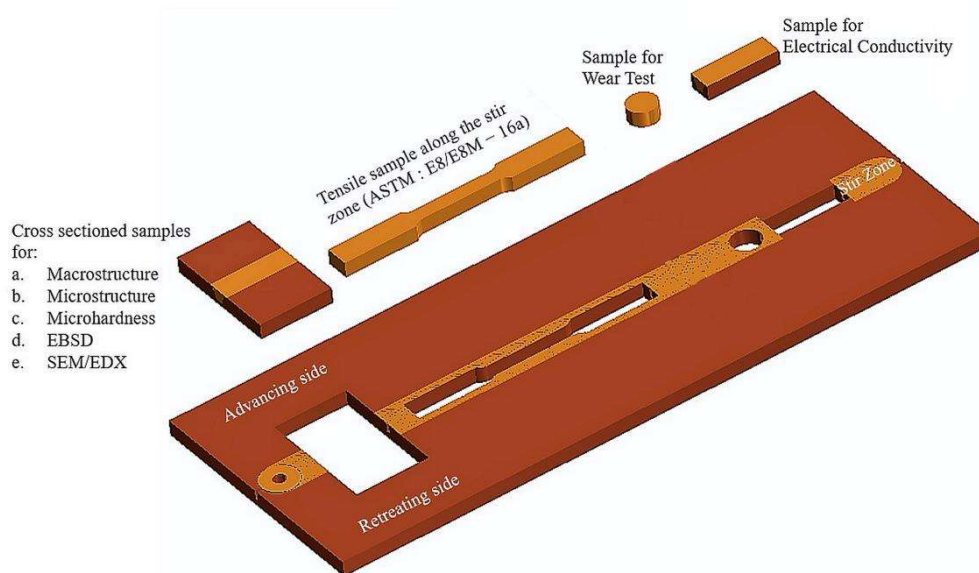


Figure 3. 6 Schematic diagrams for sample extraction from the processed plates

The microstructural features were observed by optical microscopy, scanning electron microscopy and electron back scattered diffraction technique whereas, the compositional analysis was performed through X-ray diffraction. The various tests performed to assess the performance of the composites were microhardness, tensile testing, unidirectional pin on plate dry sliding wear test, four probe technique for electrical conductivity and leaching test for the environmental impact of Cu/FA composite.

3.5.1 Optical Metallography

Specimens for the metallographic study were machined randomly, perpendicular to the FSP direction from the SZ of the FSPed plates with and without reinforcements by the abrasive cutter. Specimens were prepared according to the standard metallographic procedure (ASTM E3-11) i. e. grinding (SiC emery papers), mechanical polishing (with diamond paste) and chemical etching (10 g chromic acid, 1 g sodium sulphate, 0.85 ml hydrochloric acid and 100 ml distilled water). The microstructure of the prepared samples was observed under optical microscopy (Serial no.-DEW/101, DEWINTER Technologies, Italy).

3.5.2 Scanning electron microscopy (SEM)

The samples for SEM analysis were also extracted from the SZ of the processed plate with reinforcements exactly in the same manner as it was taken out for optical microscopy. The dimensions of the specimens were 10 mm x 10 mm x 10 mm. Specimens were first polished on different grades of emery papers (400-2000 grit size) and then it was mechanically polished with diamond paste. The samples were not chemically etched as it was done in case of optical microscopy. The mechanically polished samples were observed on SEM (Serial no.- EVO18-20-45, ZEISS EVO 18 RESEARCH, Germany).

The fracture behaviour of the samples resulting from tensile testing was characterized using SEM. Wear surfaces of base copper, processed with and without reinforcements resulting from the pin on plate unidirectional dry sliding wear tests were also analyzed for wear mechanism by SEM.

3.5.3 Electron back scattered diffraction (EBSD)

The microstructural study and grain size distribution of base copper, copper processed with and without reinforcements were performed by EBSD. The metallographically prepared samples were electro polished in a mixture of perchloric acid and methanol at -12 °C and 11 V for EBSD analysis. EBSD observations were made in an FEI FEG SEM equipped with TSL-OIM software using a step size of 250 nm.

3.5.4 X-ray diffraction (XRD)

X-ray diffraction study was carried out for compositional analysis by using diffractometer (Model- MINIFLEX 600 DETEXULTRA, Japan). Cu-K α radiation of wavelength 1.5402 Å with Ni filter was used.

3.6 Microhardness

Vickers microhardness testing machine (Model- SEMIAUTOMATIC, Serial no.- 104, MICROMACH Technologies, Pune) was used for microhardness test. Before conducting the test, samples were polished carefully so that the microstructure can be visualized in the microscope enclosed with the machine. A load of 500 gram was applied for 10 s to carry out the Vickers hardness. The test was carried out along a transverse section (perpendicular to the processing direction) which was 1 mm below the top surface. The spacing between consecutive indentations was 0.5 mm. Three hardness readings were taken for one hardness value along the same plane.

3.7 Tensile test

The mechanical strength of base copper and processed copper with and without reinforcements were evaluated by utilizing an Instron electronic tensile machine at ambient temperature condition at a strain rate of $1 \times 10^{-3} \text{ s}^{-1}$. Tensile specimens as shown in Fig 3.7 were machined from the processed plate along the length of SZ up to a depth of processing.

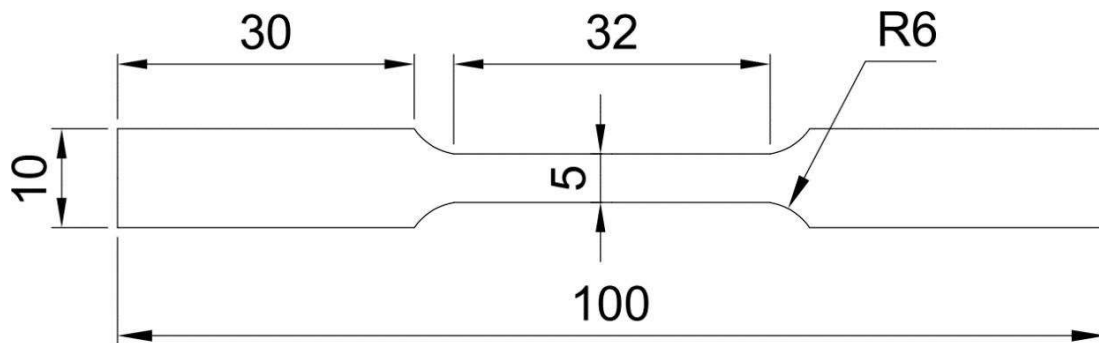


Figure 3. 7 Schematic views of the tensile specimen

For tensile specimens, the processed plate was milled by 2 mm from the backside to remove the unprocessed region and samples were extracted from the stir zone along the length of the plate by using CNC end mill cutter. The thickness of the tensile specimen was 4 mm up to the processed depth of the plate and was prepared as per ASTM: E8/E8M-16a. The properties data for each condition was obtained by averaging three test results.

3.8 Wear test

Dry sliding wear tests were performed on a pin on disk friction and wear testing machine (Model- BRO21HO, Serial no.- 116504, Magnum engineers, Bangalore, India). Schematic diagram for the wear set-up has been shown in Fig. 3.8.

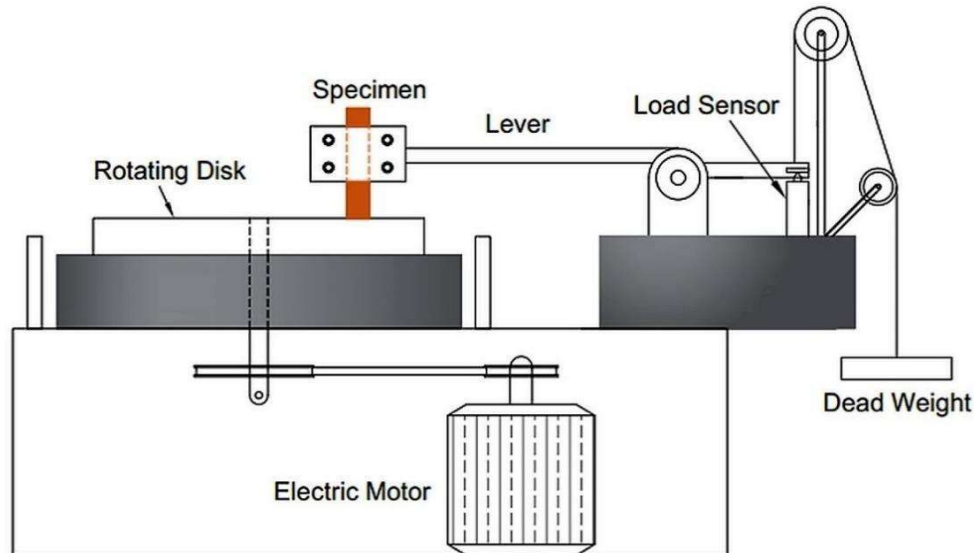


Figure 3. 8 Schematic diagrams for the pin on disc wear set-up

For wear test, samples were prepared in the form of the pin from the centre of the processed zone as shown in Fig. 3.6. The pin was in shape of a cylinder of diameter 6 mm and thickness 5 mm. A holder was fabricated to hold the wear specimens. The pin was slid against a hardened ground steel (EN-32) disk with a hardness of 65 HRC. The tests were performed on a constant load and sliding velocity of 20 N and 1m/s respectively. Before starting the test, the pin and disk surfaces were polished with different grades of emery so that maximum conformity can be established between disk and pin. Every test was conducted for 1000 m sliding distance. Wear loss was evaluated by taking the weight loss of the pin for every 200 m sliding distance. The weight loss was observed on an electronic weighing machine having accuracy up to 0.1mg. Specimens were cleaned by

acetone after each test to remove debris stuck with a pin to maintain the uniformity of the test. The properties data for each condition was obtained by averaging three test results.

3.9 Electrical conductivity

The electrical conductivity was measured with the help of Keithley nano-voltmeter (Keithley Instruments, Cleveland, Ohio, USA Model: 2182/2182A) by four probe method. The dimensions of the samples were 40 mm x 10 mm x 4 mm. The properties data for each condition was obtained by averaging three test results.

3.10 Leaching test

The environmental impact of FA and fabricated Cu/FA composite was estimated by the standard shake method for extraction of solid waste with water (ASTM-D3987). The leached solution was analyzed for metal concentration by inductively coupled plasma-spectrometer (MAKE Thermo Scientific, Model ICAP 6200 DUO and Serial no. 133226).