Synthesis and characterization of pulsed electrodeposited Cu-Y_2O_3 coating

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In the present study electrodeposited Cu-Y_2O_3 composite coatings on copper substrate were synthesized from acidic copper sulfate bath with different concentrations (10 and 30g/l) of Y_2O_3 ultrafine particles using direct and pulsed direct current mode for better surface-mechanical and oxidation properties. The study was aimed for improvement of surface-mechanical and oxidation behavior of the intended coatings compared to pure copper with retention of considerable electrical conductivity for possible electrical applications.

Phase/ morphology and elemental analysis of the coatings were carried out by XRD, SEM and EDS analysis. Surface-mechanical properties of deposited samples were studied systematically by microhardness and ball-on-plate type wear test. Thermal oxidation of the coating was also carried out. The coated samples' electrical conductivity was analyzed by four probe technique.

From the results, it was revealed that higher Y_2O_3 content in electrolyte tends to increase Y_2O_3 content in the deposits, which results in better hardness and wear resistance. Higher hardness and better wear resistance properties of co-deposited coatings were also attributed to finer matrix resulted by pulsing mode with higher pulse frequency. Better oxidation resistance was recorded in composite coatings due to better microstructure. Minor decrease in electrical conductivity of composite coatings was observed due to presence of Y_2O_3 and finer matrix, but values were in acceptable range. The present method can be considered useful to improve surface properties of electrically used copper components.

Key words: Electro-codeposition; Cu; wear; oxidation; electrical conductivity.

References:

- L. Tian, J. Xu, Electrodeposition and characterization of Ni–Y_2O_3 composite, Appl. Surf. Sci. 257 (2011) 7615–7620.
- H.S. Moharana, A. Basu, Surface-mechanical and oxidation behavior of electro-co-deposited Cu-Y_2O_3 composite coatingElectrodeposition and characterization of Ni–Y_2O_3 composite, Surf. Coat. Technol. 304 (2016) 348–358.

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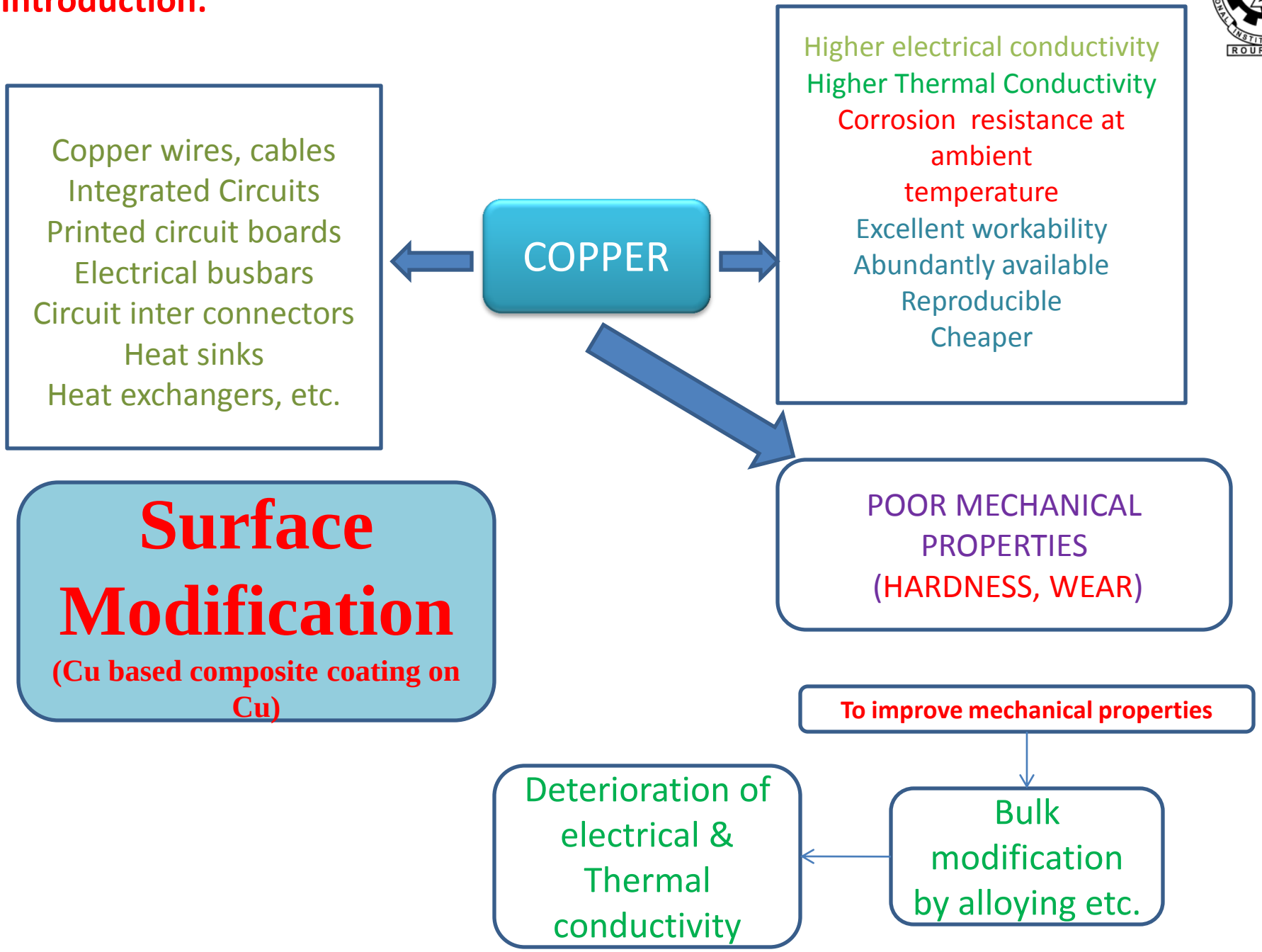
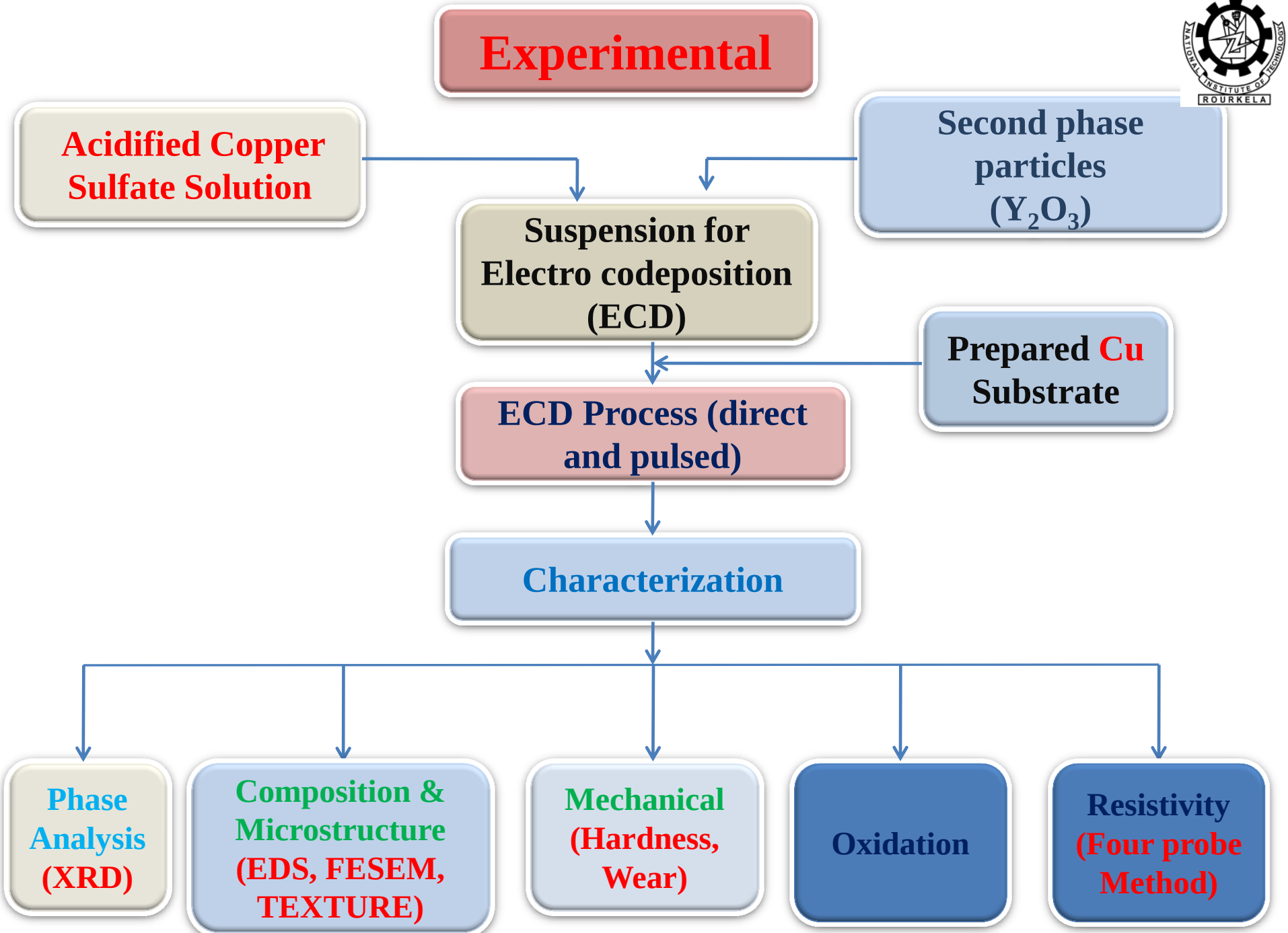
ICAMMP 2016

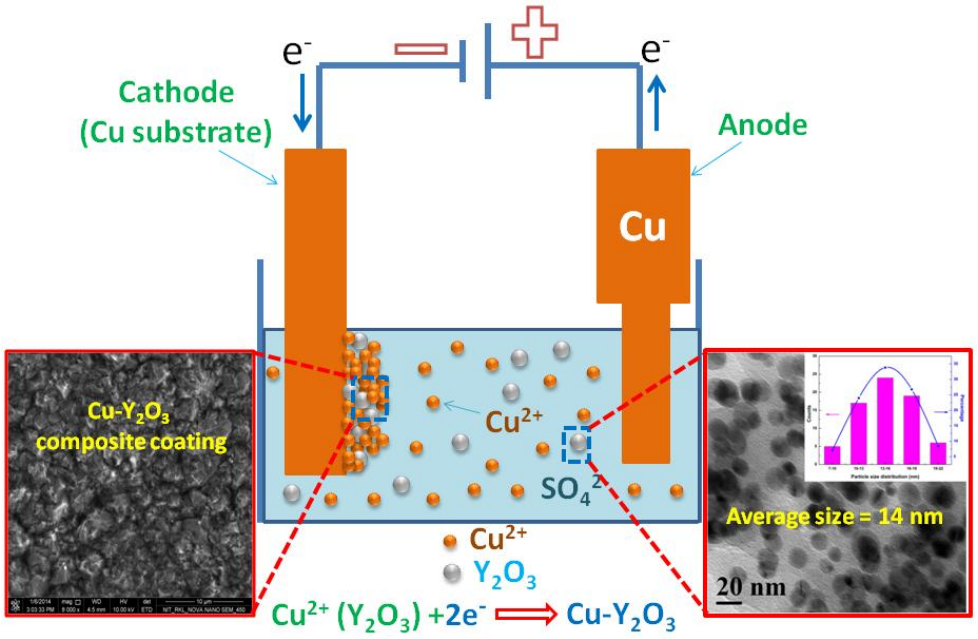
Introduction:



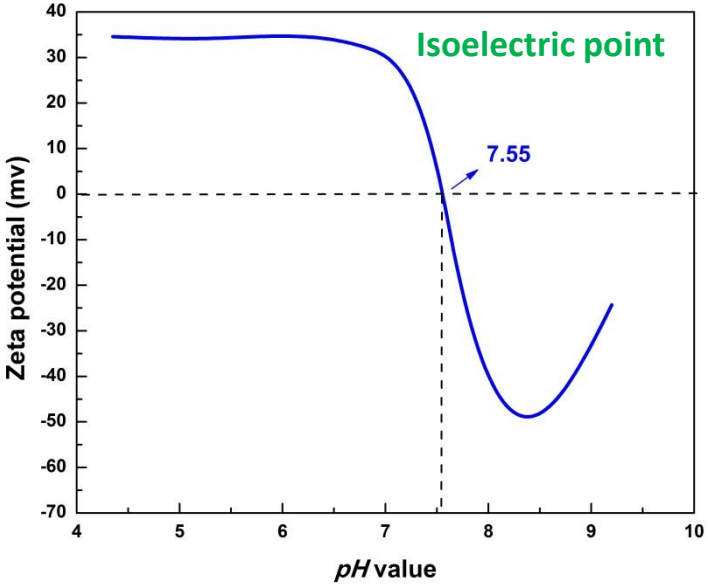

Experimental
Acidified Copper Sulfate Solution
Second phase particles (Y_2O_3)
Suspension for Electro codeposition (ECD)
Prepared Cu Substrate
ECD Process (direct and pulsed)
Characterization
Phase Analysis (XRD)
Composition & Microstructure (EDS, FESEM, TEXTURE)
Mechanical (Hardness, Wear)
Oxidation
Resistivity (Four probe Method)

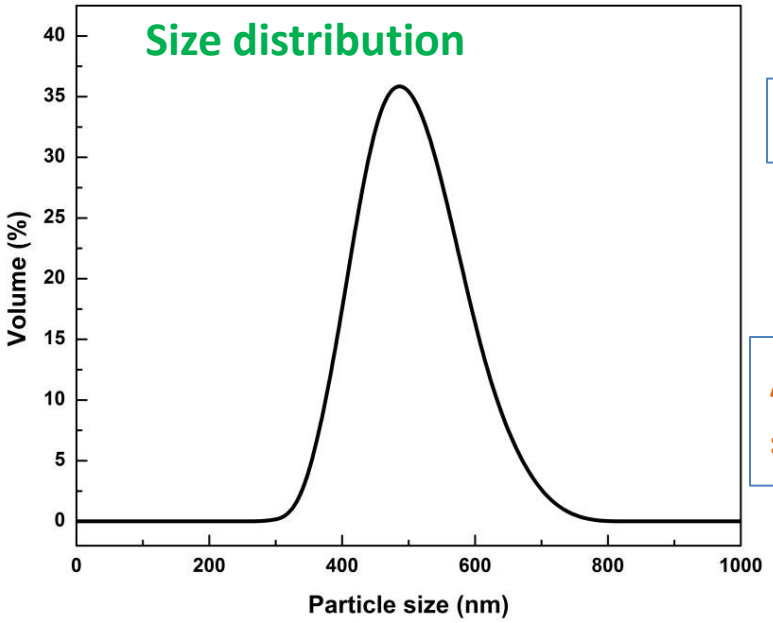
Plating bath compositions and deposition parameters and process:

Item	Details
Electrolyte	Copper Sulfate ($CuSO_4.5H_2O$): 200 gm/l, Sulfuric acid (H_2SO_4): 50 gm/l
pH	~2.17
Current density	8 A/dm^2
Temp.	Room temperature
Plating time	20 minutes
Dispersion	Y_2O_3: 0, 10 and 30 gm/l
Deposition mode	DC and Pulsed DC [pulse frequency: 1, 5 and 10 kHz and duty cycle (pulse on time):30%]



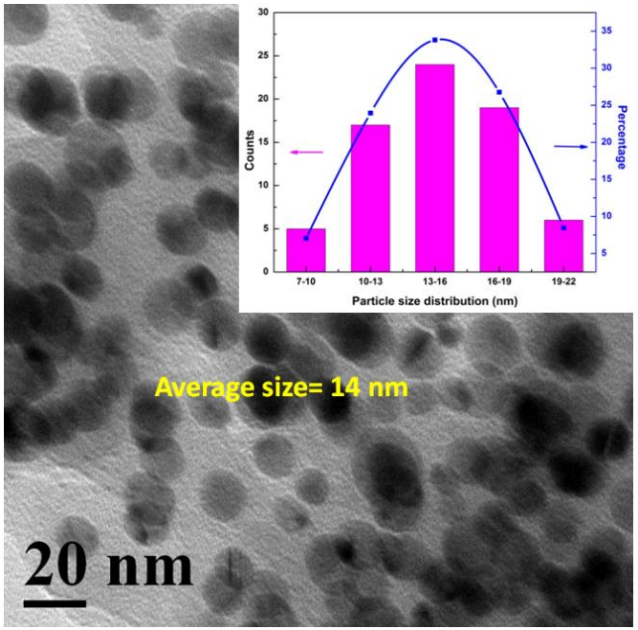
Characterization of as procured Y_2O_3 powder

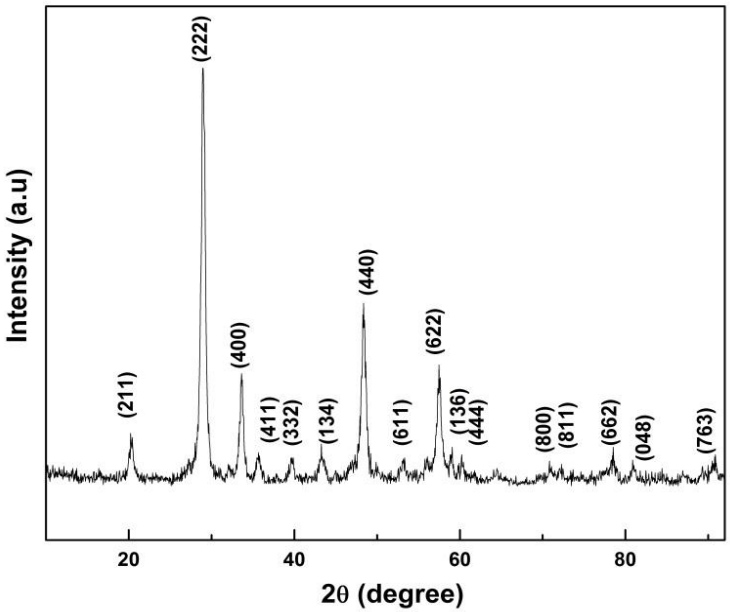




IEP=7.55

Avg. Size (Zeta) = 480nm

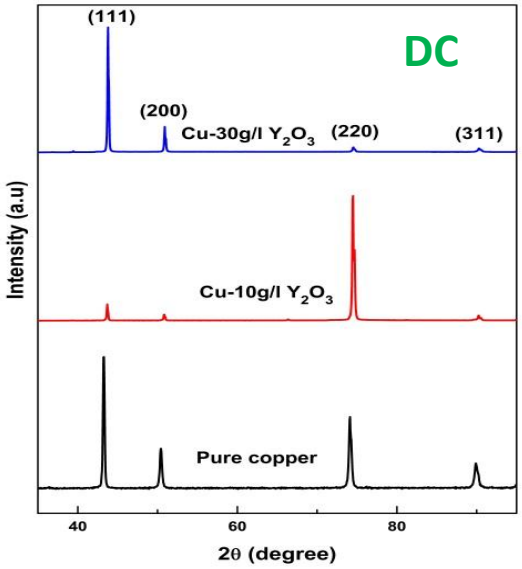


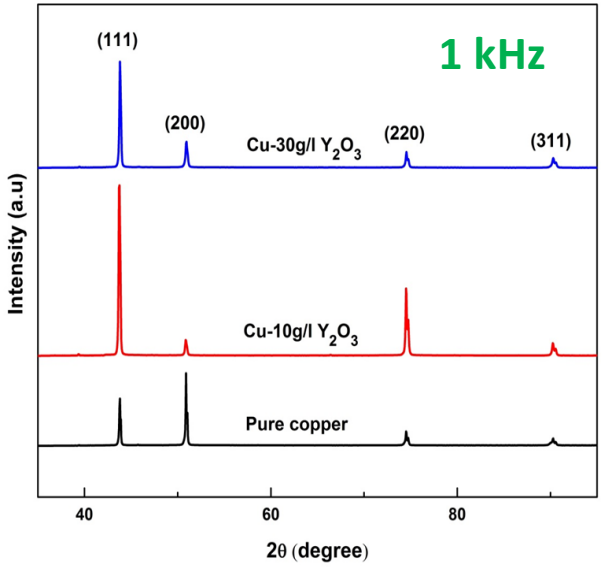


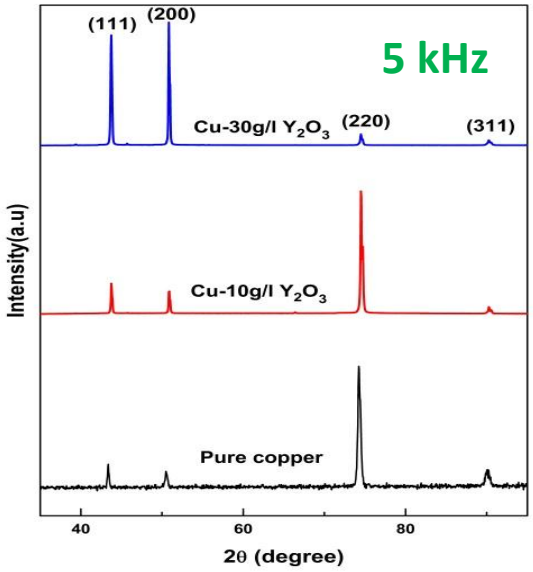
Avg. Size (TEM) = 14nm

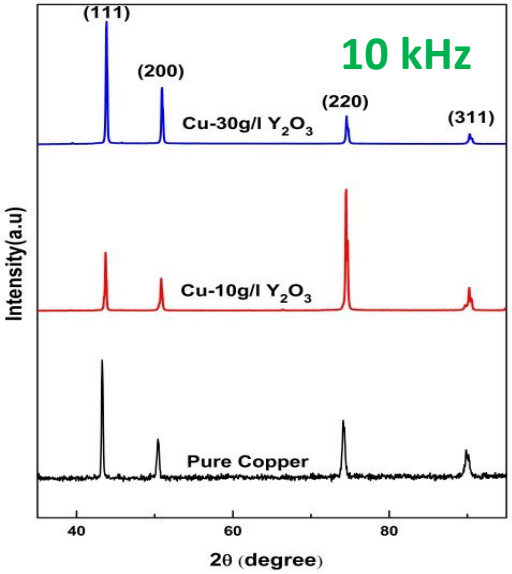
BCC Crystal structure

XRD analysis

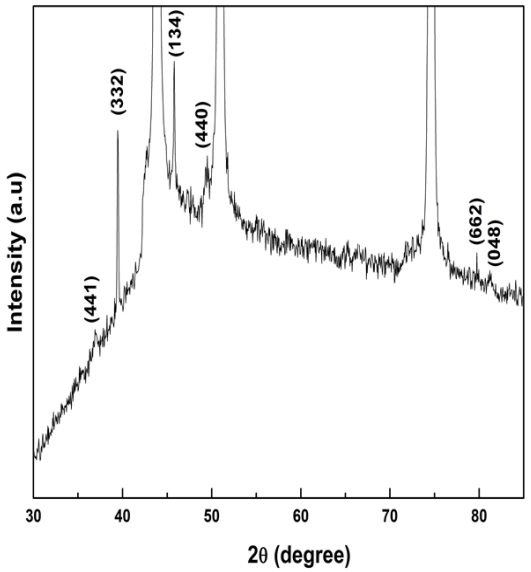








Change in relative intensities of Cu peaks at different coating conditions

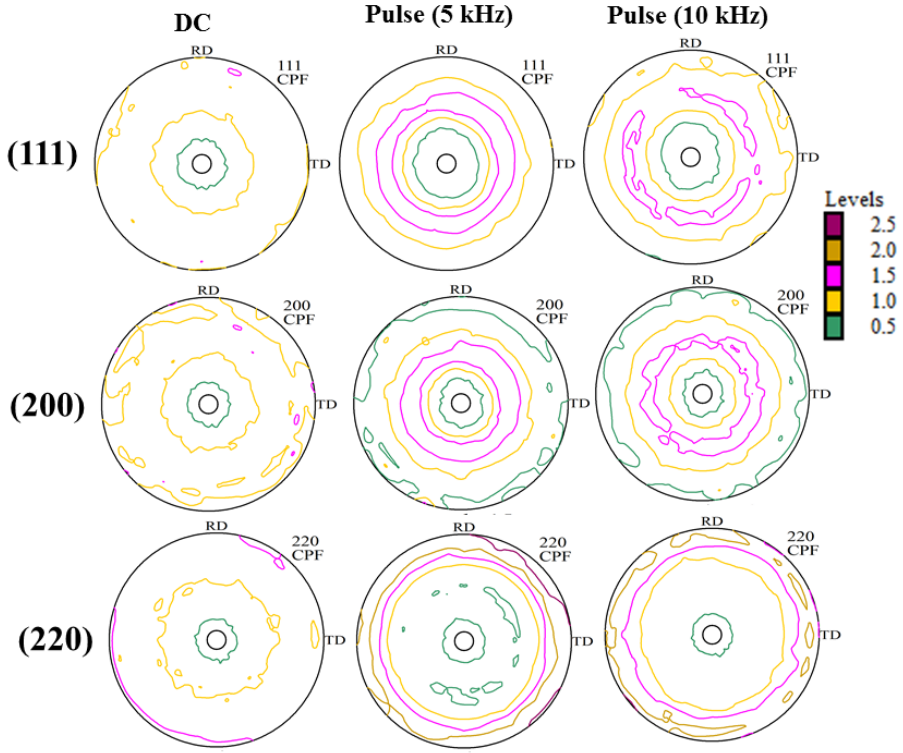


Enlarged view of XRD plot of Cu-30g/l Y_2O_3 coating with 10 kHz.

Y_2O_3 peaks are visible

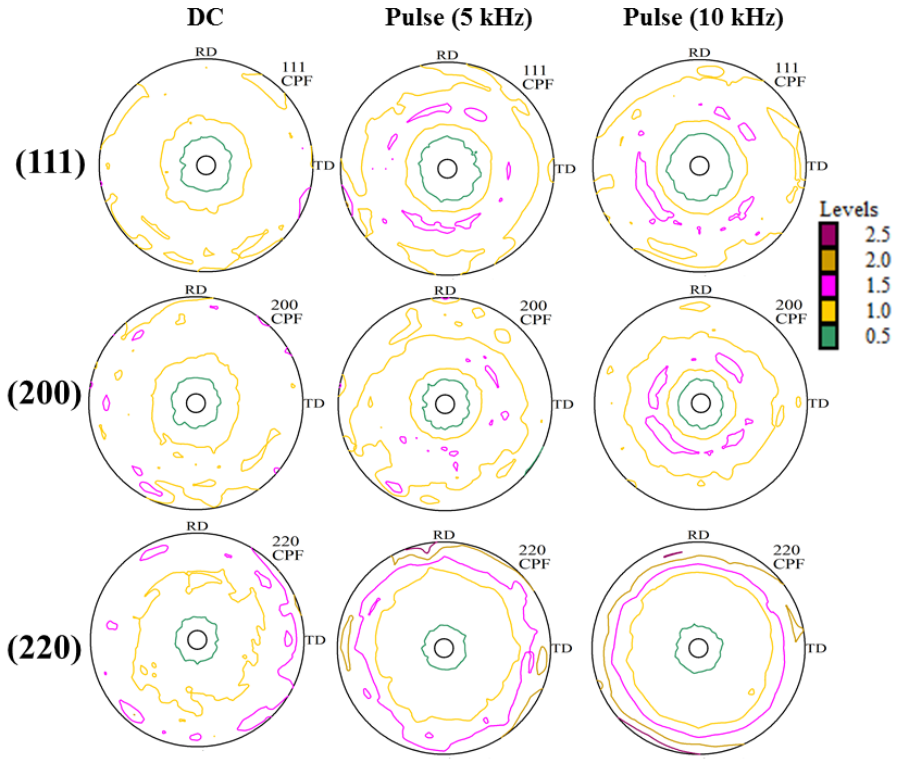
Texture analysis

Pure Cu



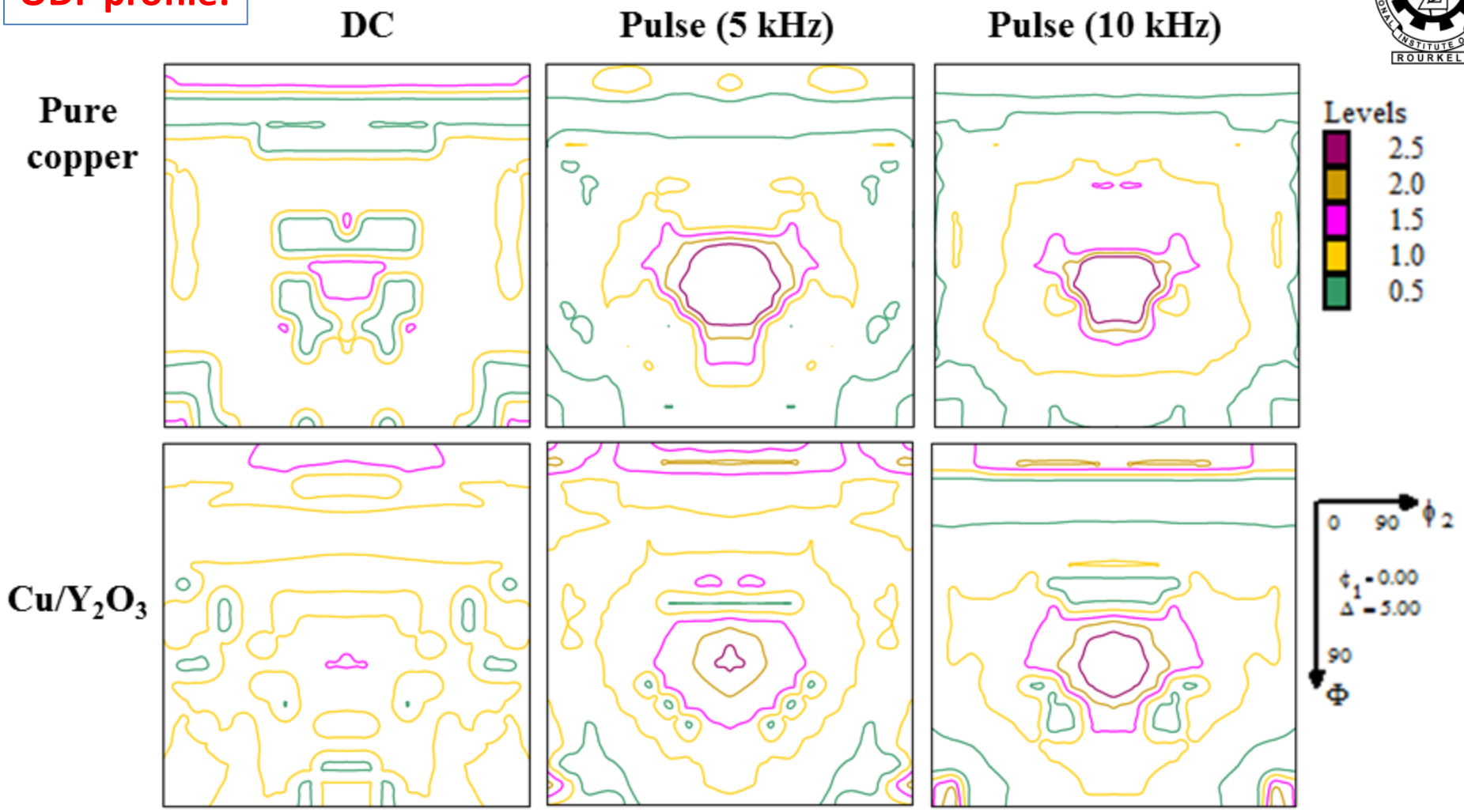
With changing deposition condition from DC to pulse frequency of 5 and 10 kHz, the initial intensity of [001], [110] and [111] orientations decreases. But the [111] orientation remained as preferred orientation

Cu-30g/l Y_2O_3



Changing deposition condition from DC to 10 kHz pulse increases [111] fibre orientation and decreases other orientations ([001] and [110])

ODF profile:

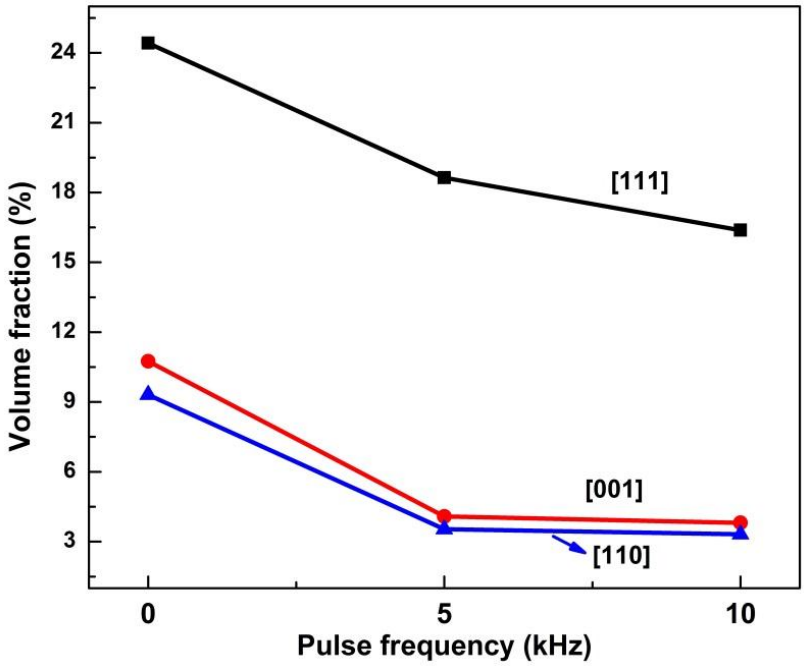


ϕ_1= constant ODF figure of pure copper and Cu-30g/l Y_2O_3 composite coatings

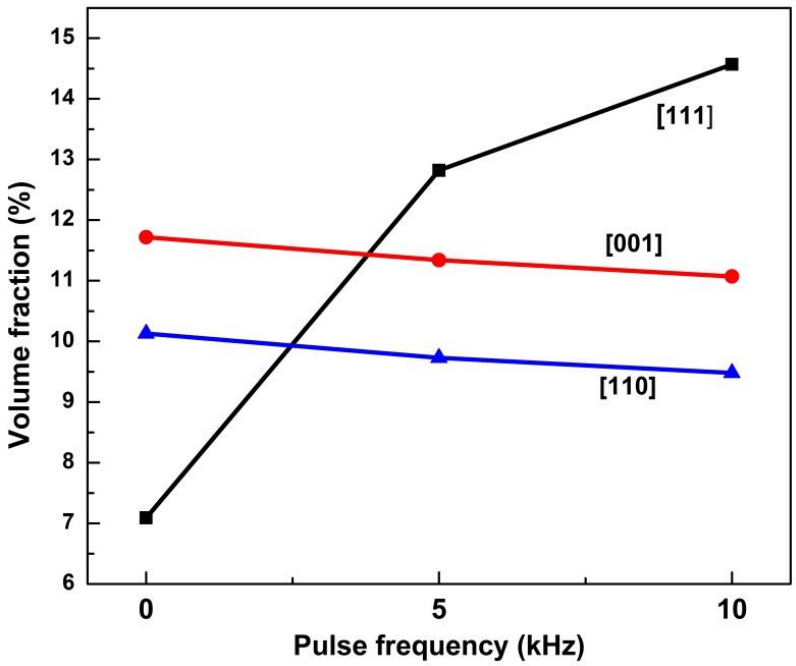
Existence of mainly [111] orientation at 10 kHz

Orientation Volume fraction:

Pure Cu



Cu-30g/l Y_2O_3

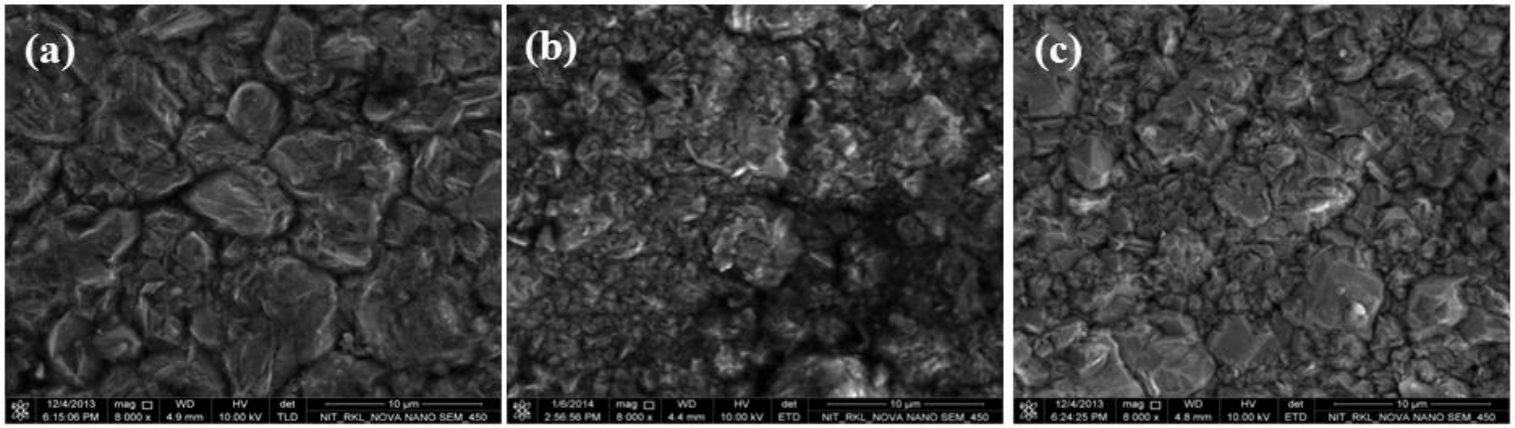


Volume fraction of different orientation confirms [111] as the main and preferred orientation. Y_2O_3 addition changes the trend of orientation may b due to impact on nucleation

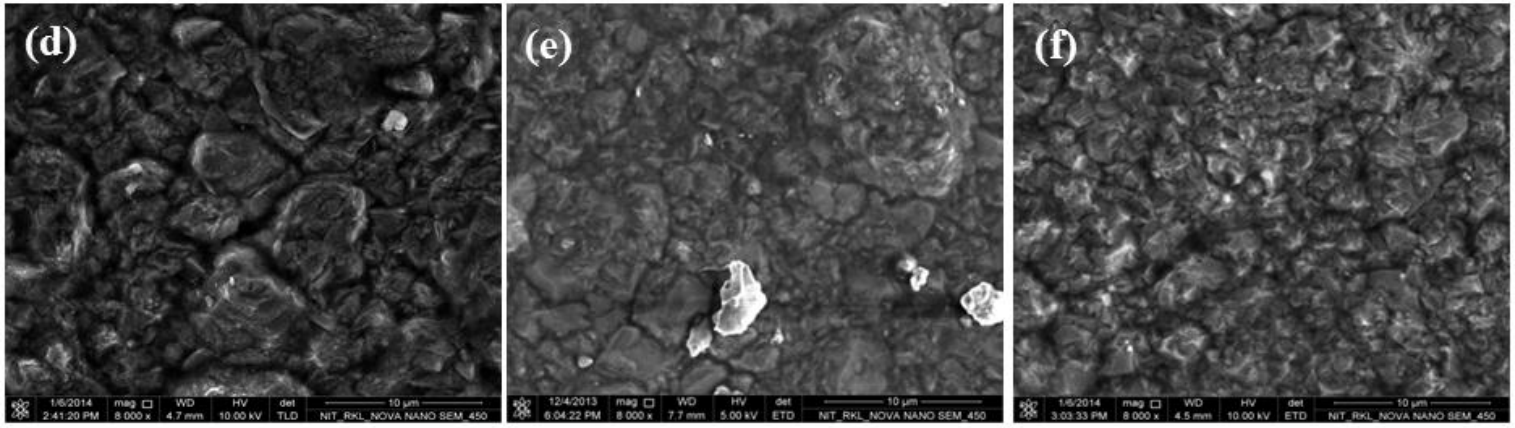
Morphology study:

DC | 5 kHz | 10 kHz

Cu-10g/l Y_2O_3



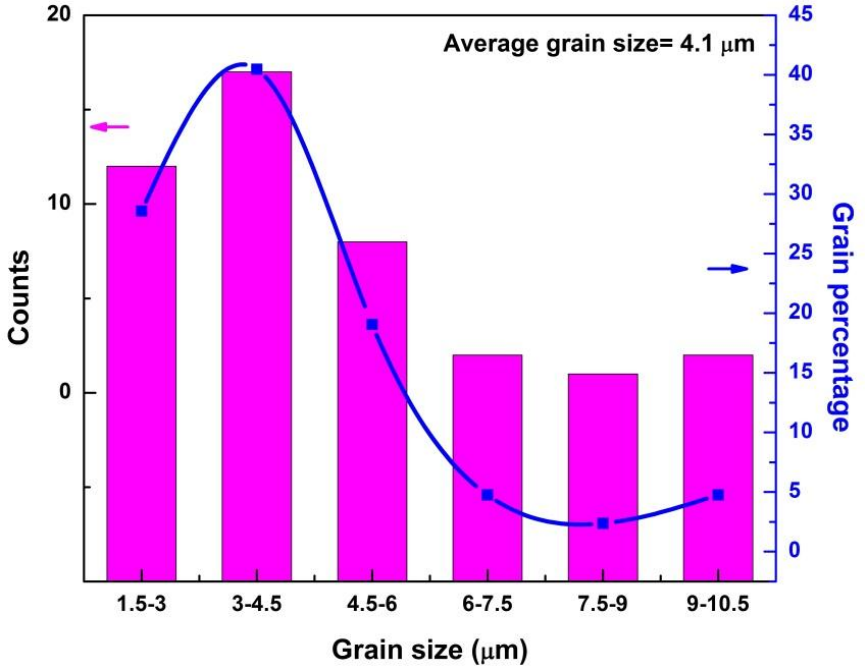
Cu-30g/l Y_2O_3



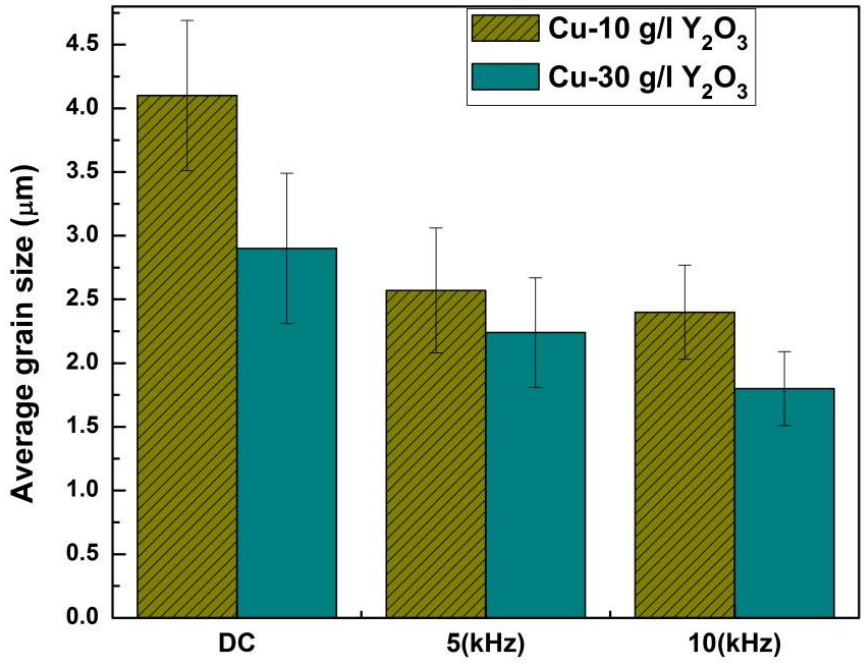
- DC deposition shows large granular structure with well-defined grain boundaries
- The coating deposited with highest pulse frequency (10kHz) exhibited well-refined granular structure with more uniform and finer matrix.

Size distribution and average grain size

Cu-10g/l Y_2O_3 (DC)



Average grain size



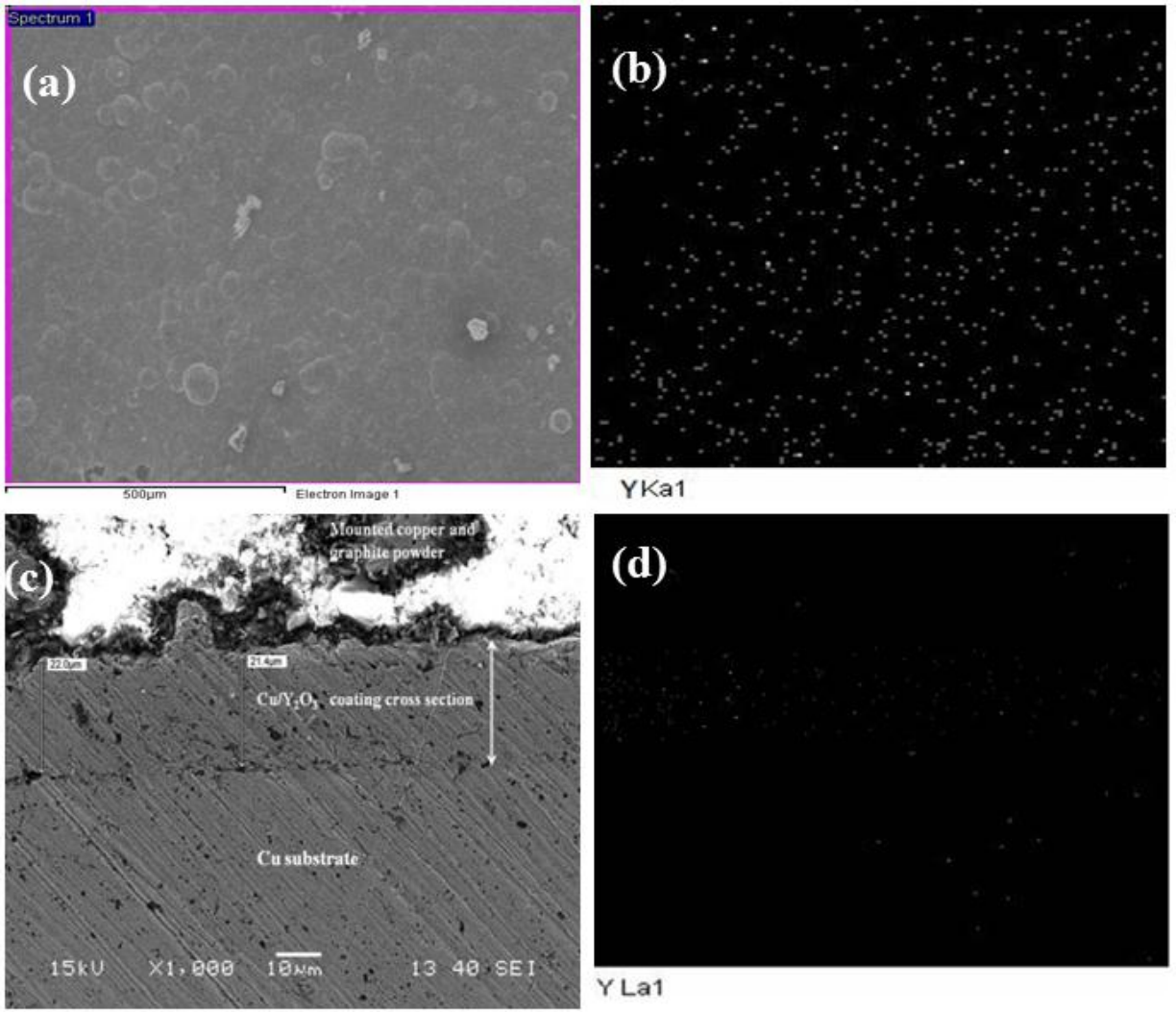
Decrease in average grain size with increase in second phase particle dispersion and pulse frequency

Published in: *Surface and Coating Technologies, 304 (2016) 348–358; Basu et. al*

Elemental mapping

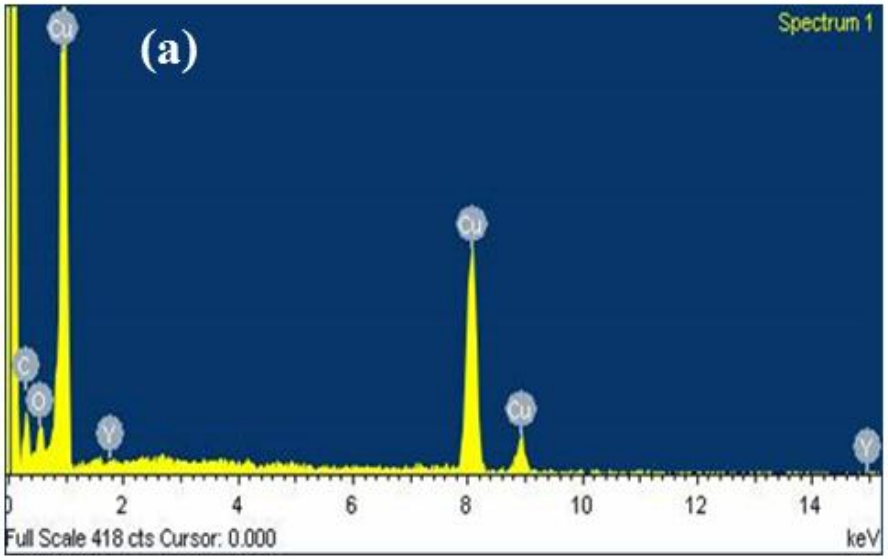
Cu-30g/l Y_2O_3 (10 kHz)

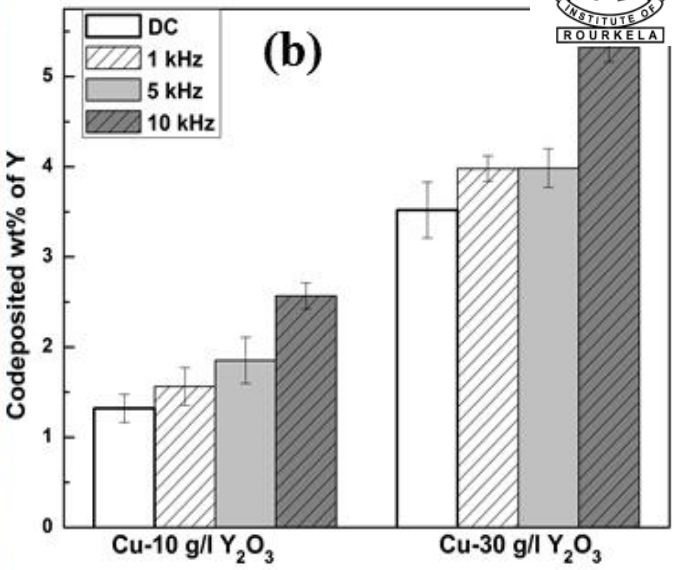
Uniform deposition of Y_2O_3 throughout the coating and cross-section



EDS spectra

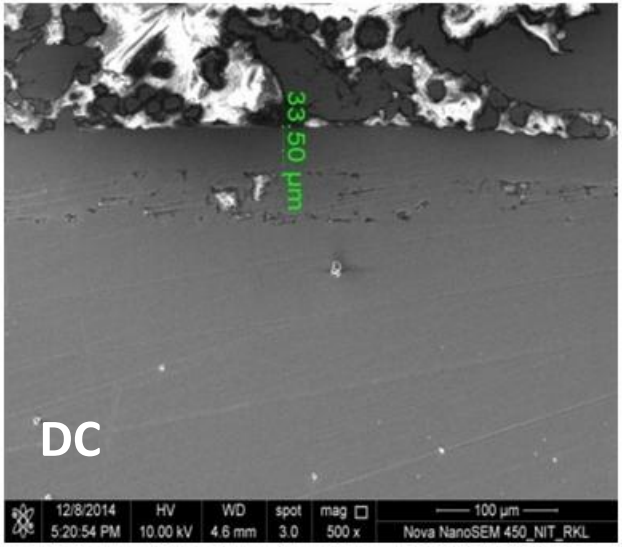
Cu-30g/l Y_2O_3(10 kHz)

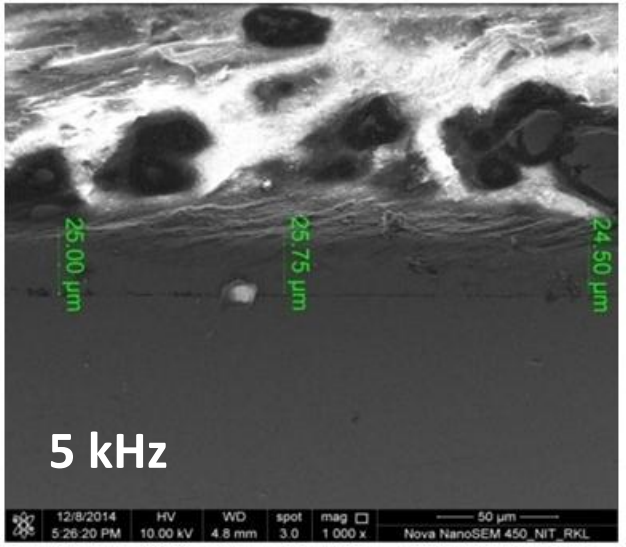




- Wt.% of Y_2O_3 in the coating increases with pulse frequency and Y_2O_3 dispersion.
- Thickness decreases with increasing pulse frequency.

Cu-30g/l Y_2O_3

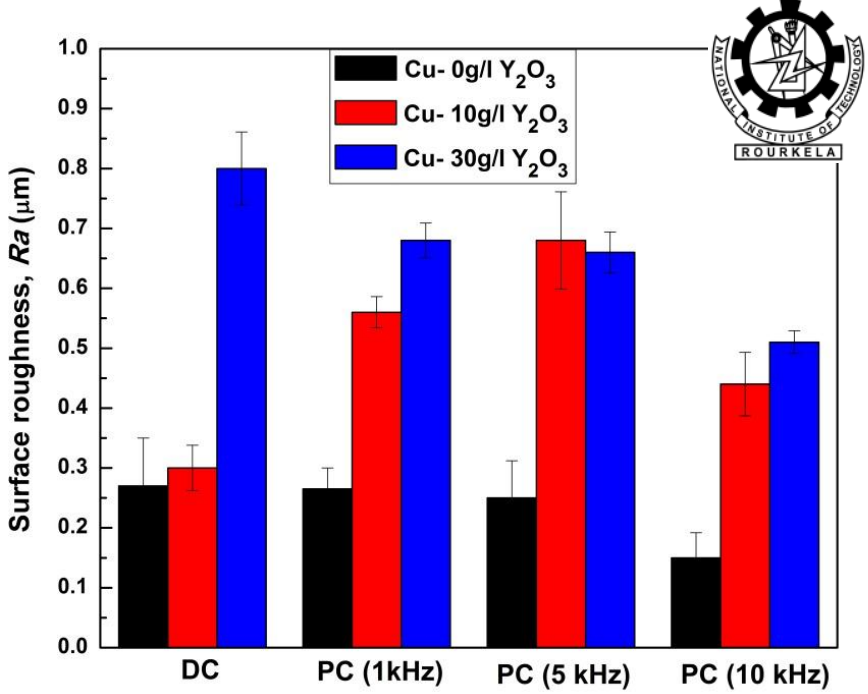




Surface Roughness

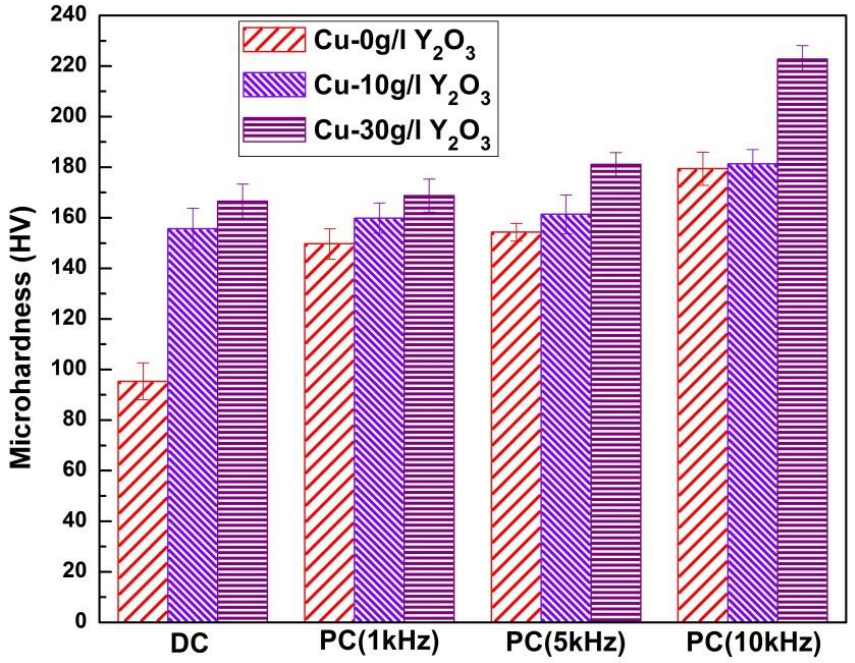
- R_a 0.14 to 0.8µm

- Surface roughness increases with increasing Y_2O_3 content except 5 kHz



Microhardness

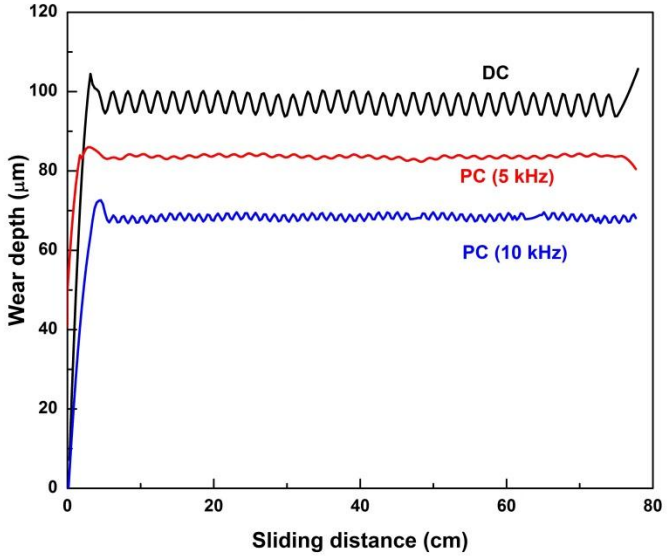
Hardness increased with increasing Y_2O_3 content as well as pulse frequency.



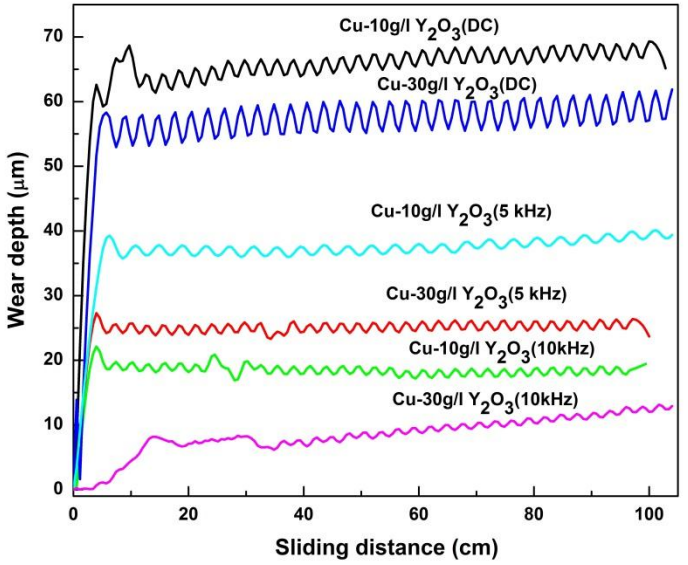
Wear study

- Wear resistance improvement by pulsing and Y_2O_3 content

Pure Cu

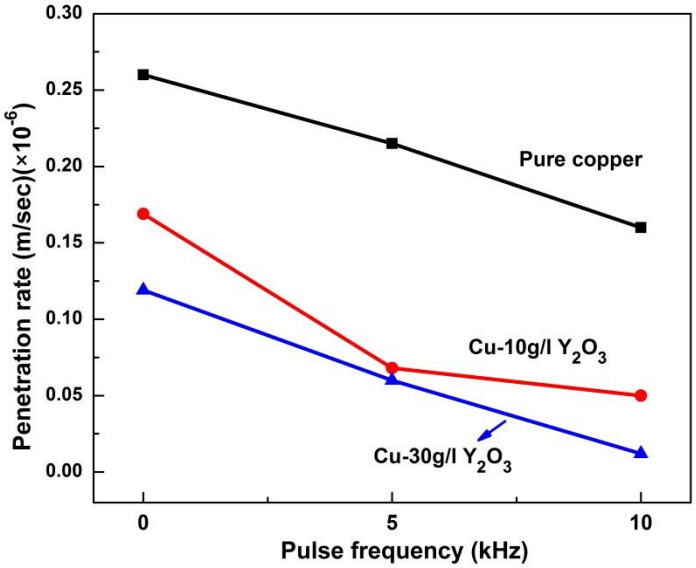


Cu-Y_2O_3

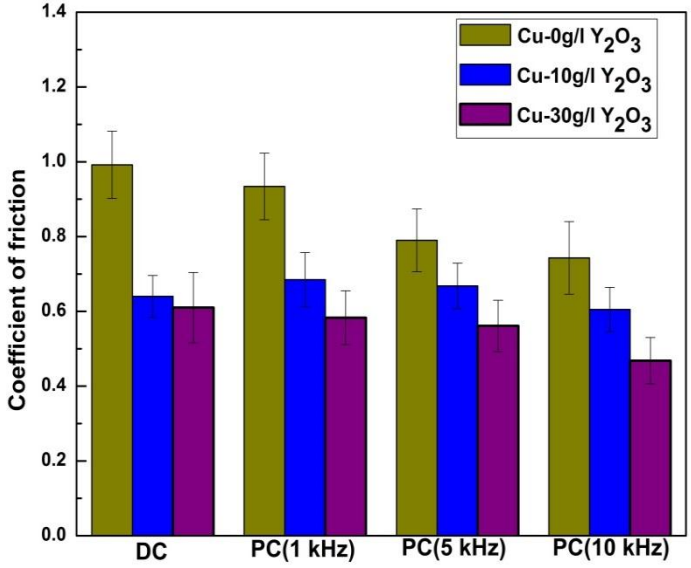


- Friction coefficient is decreases with Y_2O_3 content and pulse frequency

Wear rate

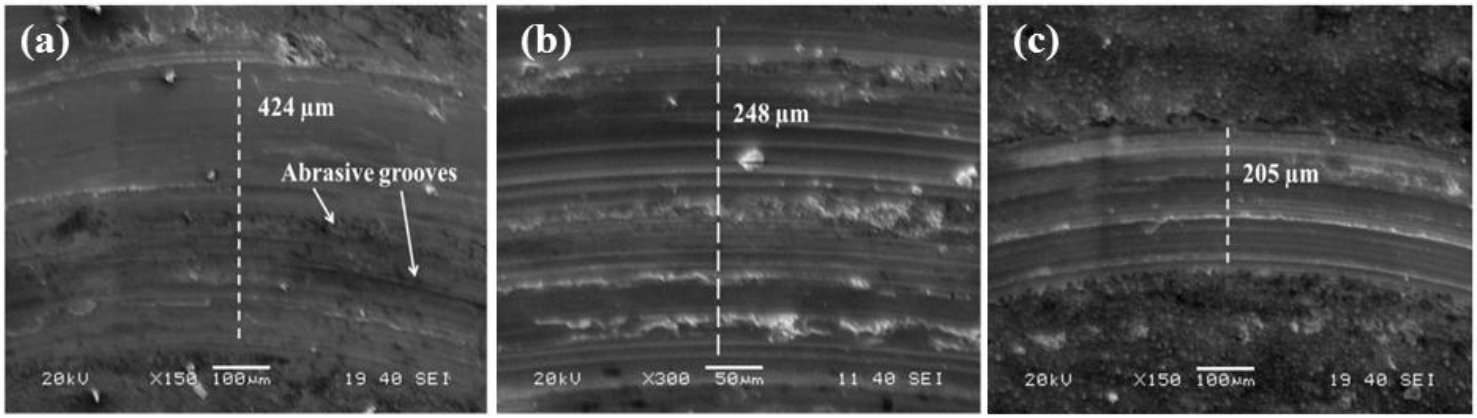


Coefficient friction



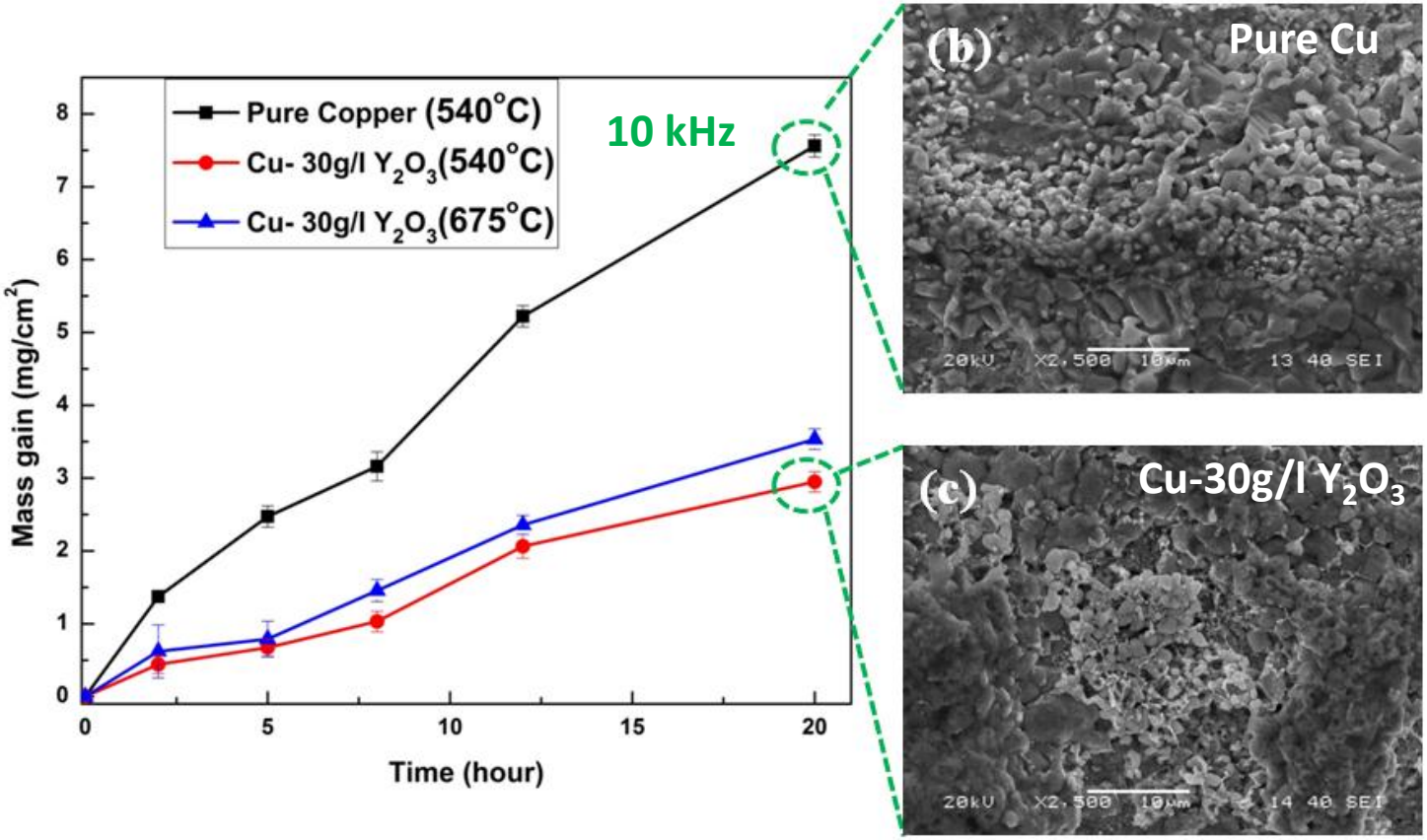
Wear track

Pure Cu (10 kHz) | Cu-10g/l Y_2O_3 (10 kHz) | Cu-30g/l Y_2O_3(10 kHz)



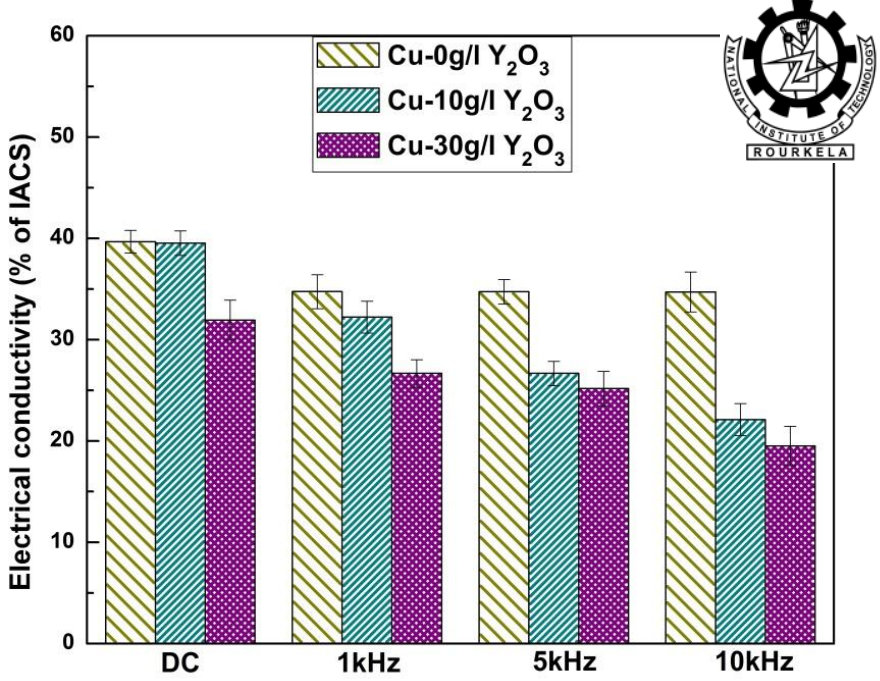
Oxidation study

- Composite coating exhibited better oxidation resistance



Electrical conductivity

- Decrease in conductivity with increase in pulse frequency in spite of (111) orientation development; can be attributed to dispersed Y_2O_3 particle, finer grain size.



After oxidation

Pure Cu- (13-24 %) ↓

Cu-Y_2O_3- (1-7.7 %) ↓

Conclusions

- Texture study confirms [111] as the main fibre orientation.
- Higher hardness and better wear resistance properties of codeposited coatings were attributed to finer matrix and more amount of embedded Y_2O_3 particles resulted by pulsing mode with higher pulse frequency conditions as well as some texture effect.
- Better oxidation resistance was found in composite coatings due to better microstructure.
- Minor decrease in electrical conductivity of composite coatings was observed due to presence of Y_2O_3 and finer matrix.

Acknowledgement

Council of Scientific & Industrial Research, India (Grant No. 22/0563/11/EMR-II).

Thank You!