

Objective:

The main objective of this work is to develop processing strategies for seamless Nb tube that give uniform through thickness microstructure and desired mechanical properties that are suitable for hydroforming.

Problem:

Obtaining polycrystalline Nb with a fine grain sizes and uniformity is a challenge [1-4].

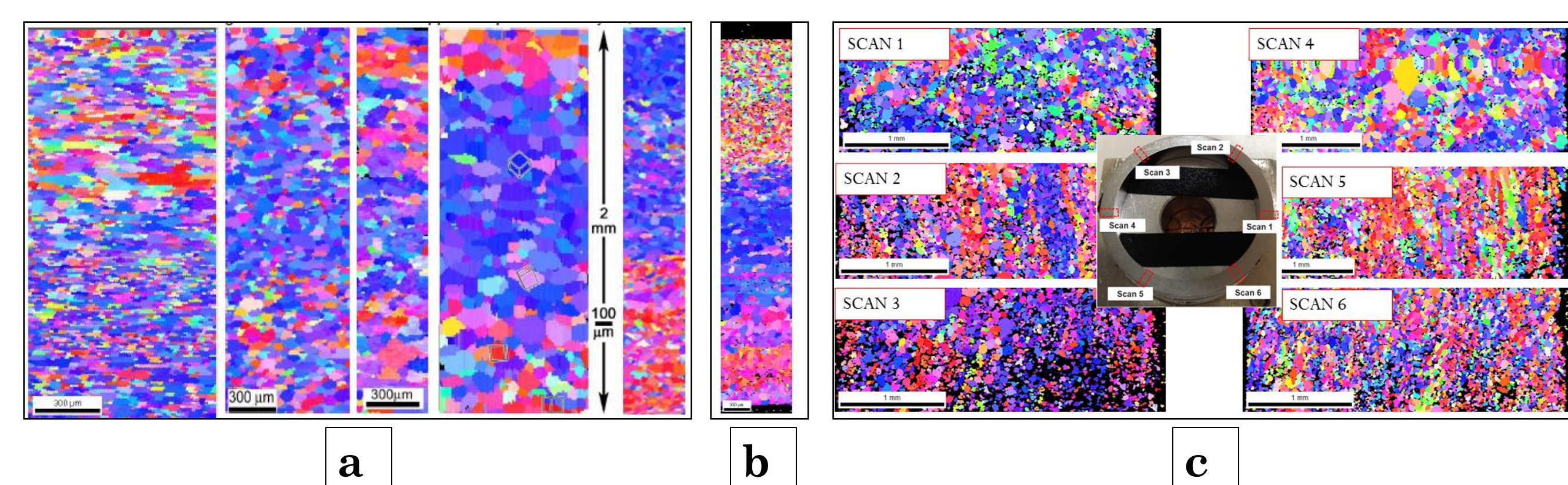


Figure 1. Inverse pole figure maps along the radial direction of the tube. a) rolled sheet material, b) back extrusion and flow forming, and c) forward extrusion/ SFI, TAMU.

Approach:

- Multi pass ECAE of Nb with intermediate heat treatments for microstructure engineering of initial Nb.
- Seamless Nb tubing with fine grain Nb microstructures.
- Microstructure modifications by Symmetric shear processing and heat treatments for circumferential uniformity.

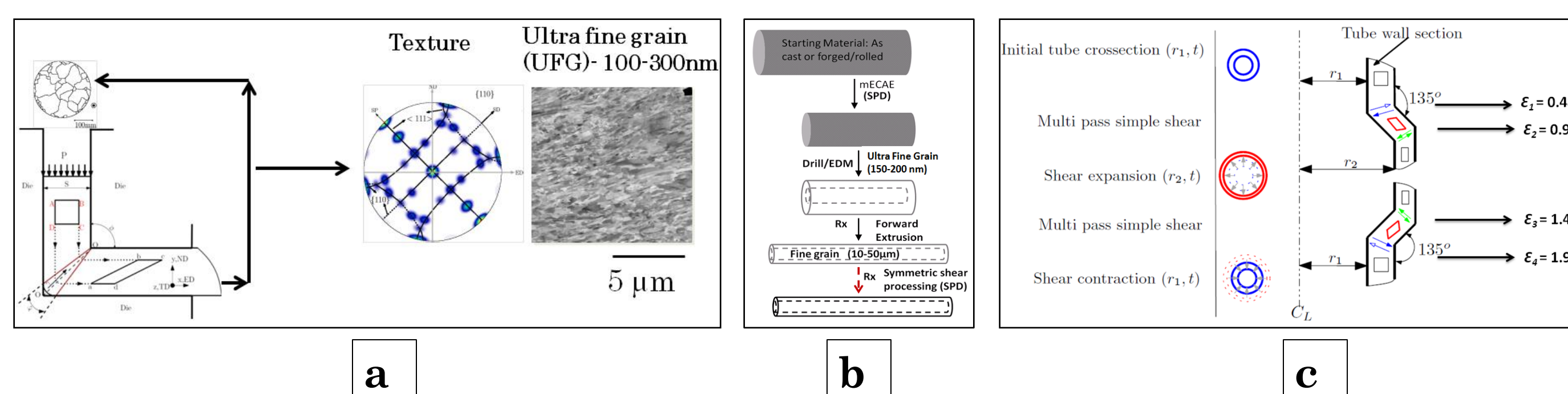


Figure 2. Approach developed to obtain circumferentially uniform microstructure in seamless Nb tube, a) Schematic of shear processing by ECAE, b) Flowchart of the steps to obtain a fine grain Nb tube, c) Schematic of tube ECAE (tECAE)

Materials and Methods:

- Material: RRR 180 Nb.
- Heat treatment: 1173K, vacuum ($<8 \times 10^{-6}$ torr), 2hr.
- Processing: ECAE, Area reduction extrusion, tECAE.
- Tensile Testing.

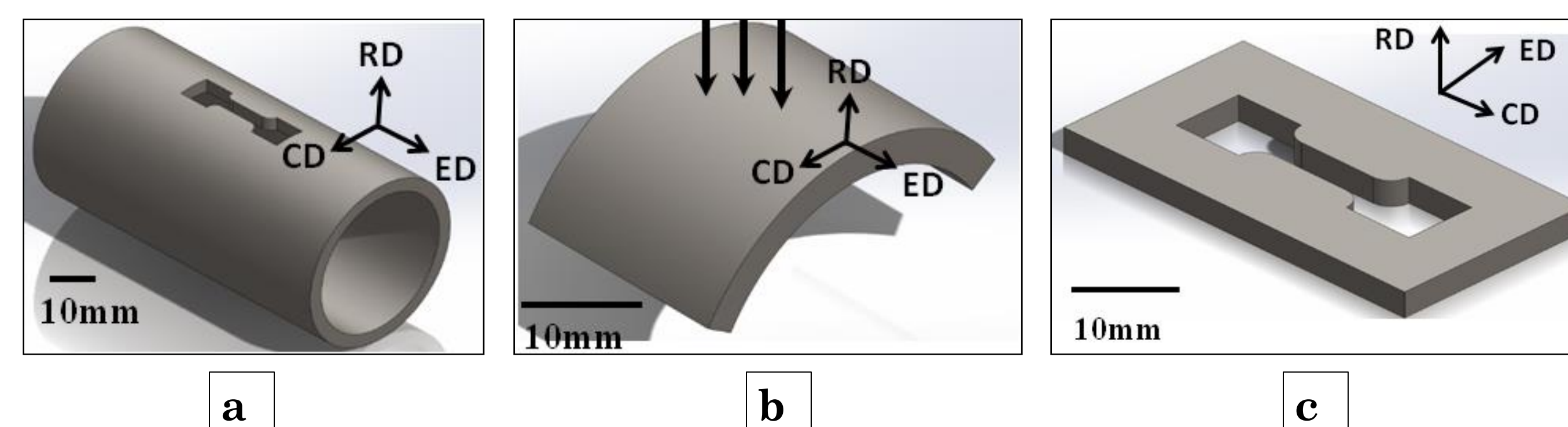


Figure 3. Schematic of the cutting plan for the tensile test samples: a) Extrusion direction (ED) sample, b) circumferential direction (CD), and c) tensile specimen cut along CD after flattening. The LD direction corresponds with the extrusion direction in all cases. RD represents the radial direction

Results and Discussion:

1. Microstructure characteristics of seamless Nb tube

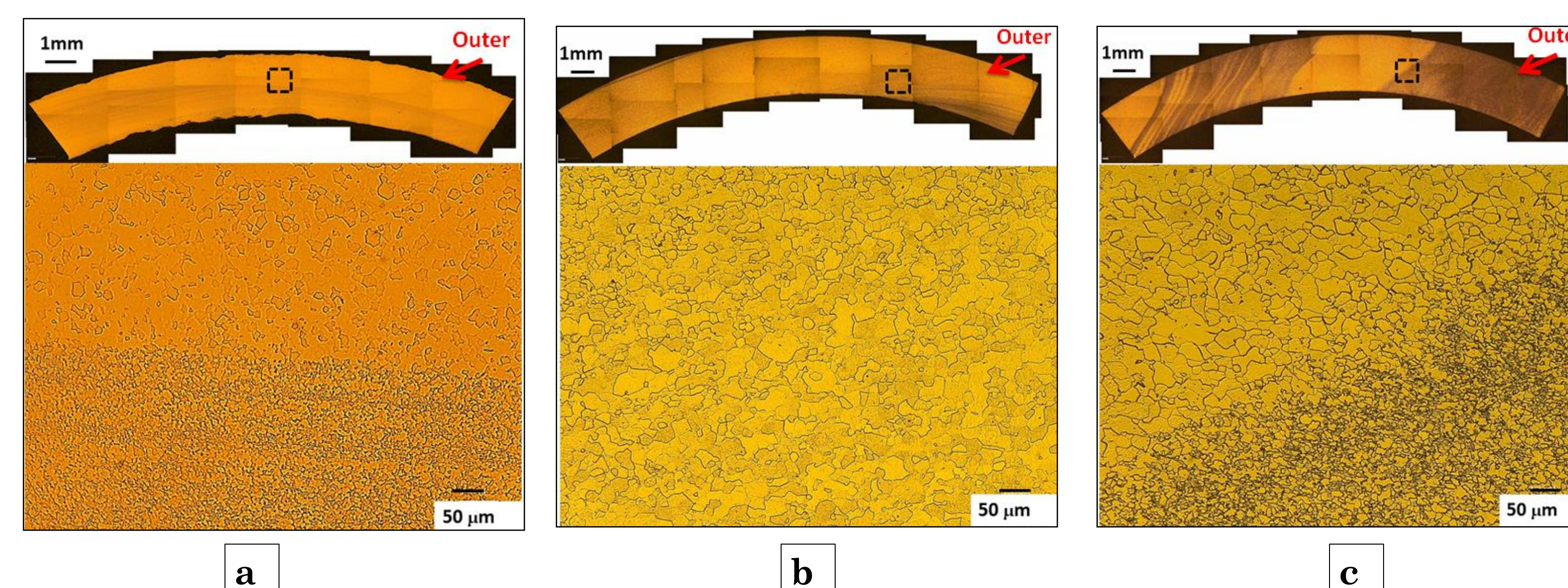
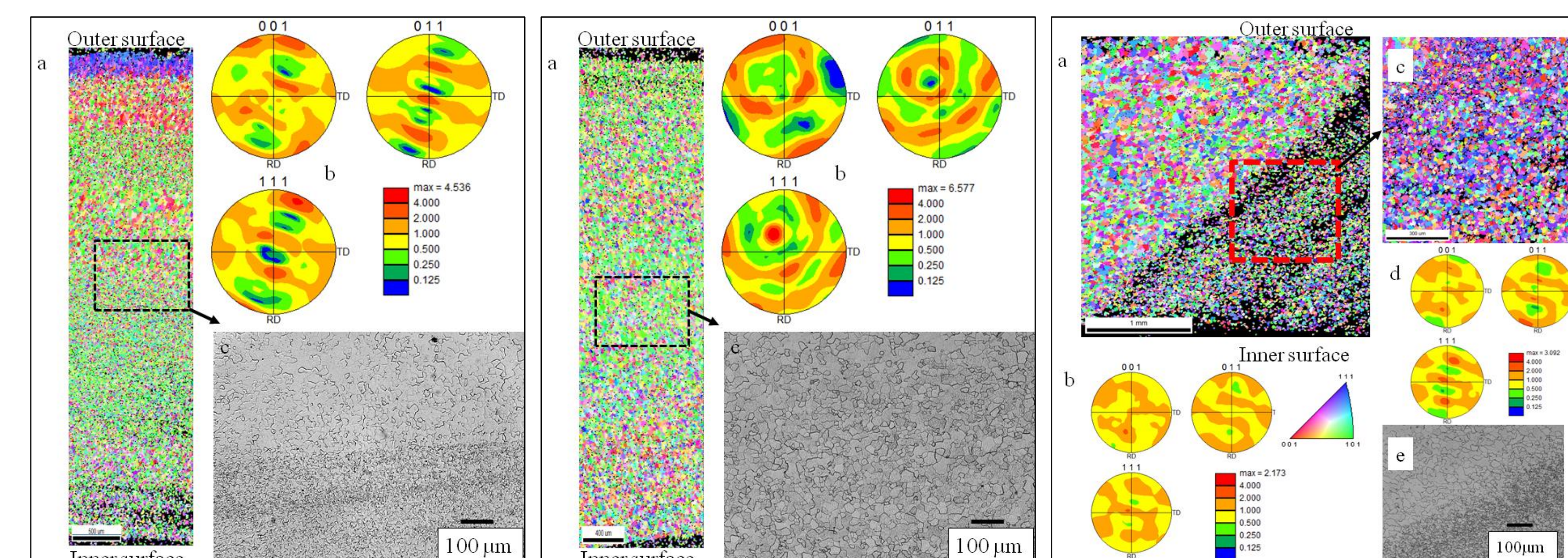


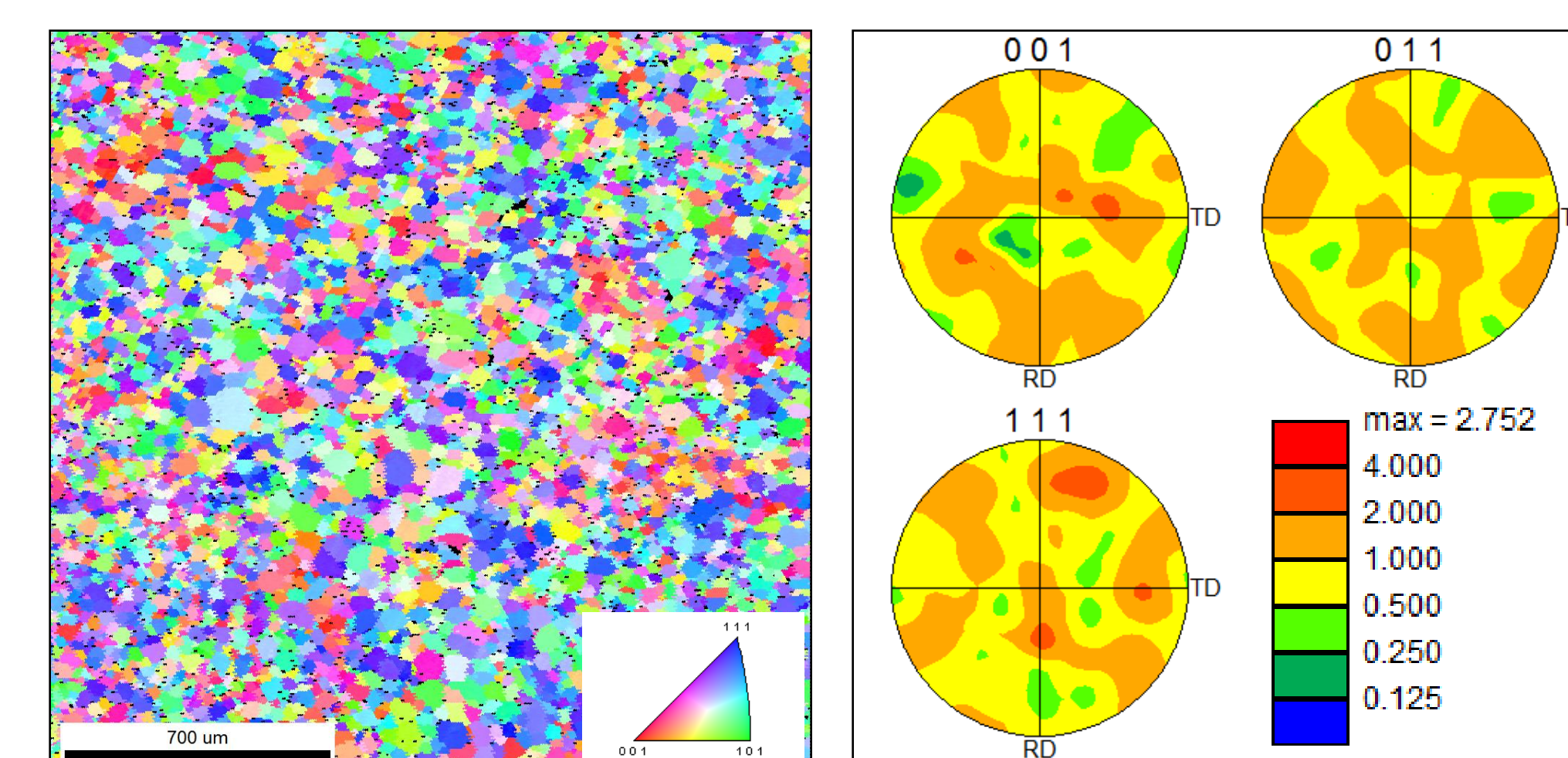
Figure 4. Representative macrograph after recrystallization at 900°C. a) area reduced tube, b) shear processed tube with no intermediate annealing, and c) shear processed tube with intermediate annealing



- Predominant $\{111\}$ | ED up to a depth of 200 μm , transitions to $\{101\}$ | ED, and $\{001\}$ | ED to a depth of 600 μm .
- Major texture component $\{001\}<011>$, and $\{111\}<011>$ spread in the RD.
- Texture components similar to b.c.c rolling textures.
- Maximum texture strength varied between 4.5-7.5 m.r.d
- No major texture variation along circumference.
- Grain size: 15 \pm 7 μm .
- Grain size banding diffuse, no geometric correlation to area reduction extrusion

- No variation in texture across the cross-section, indicating homogeneous deformation.
- $\{001\}<011>$ texture removed leading to development of a $<111>$ fiber, 35° inclined from ED towards RD.
- Maximum texture strength varied between 6.6-7.8 m.r.d
- No major texture variation along circumference.
- Grain size: 14 \pm 6 μm
- No presence of grain size banding in the areas examined.

- Large banding in microstructure.
- Emergence of a weak $\{111\}$ | ED texture.
- Textures developed are very weak. Maximum strength varies between 2-3 m.r.d along the circumference.
- Textural banding is absent.
- Grain size: 27 \pm 14 μm , 11 \pm 6 μm
- Bands stretch across the circumference from outer to inner surface of tube.
- Banding could be due to instability due to low yield strength.



- OIM map in the ED for ECAE processed pre cursor material.
- The texture observed is identical to simple shear textures (see Fig. 2a.) and weak.
- Banding observed in worked material may not be due to precursor material

Results and Discussion:

2. Mechanical testing

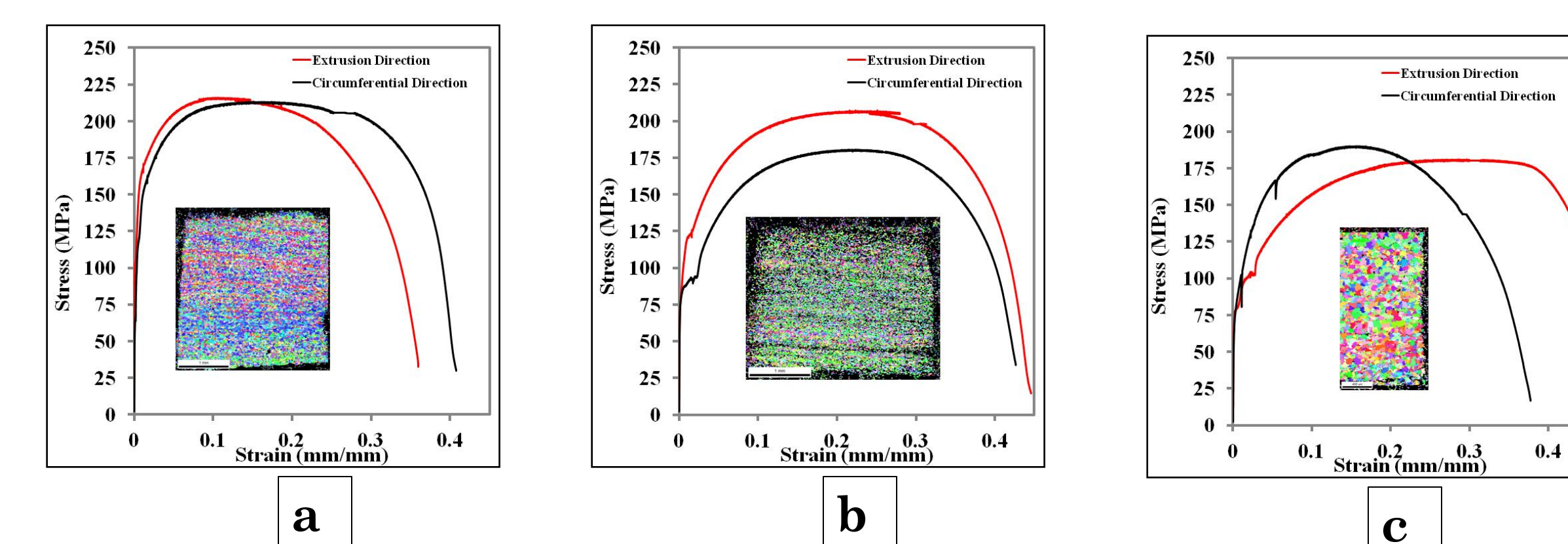


Figure 6. Tensile curves of recrystallized samples in the extrusion and circumferential directions along with the IPF map along the circumferential direction a) area reduced tube, b) shear processed tube with no intermediate annealing, and c) shear processed tube with intermediate annealing

Work Hardening behavior of polycrystalline Nb and the Holloman Equation:

Hardening behavior is quantified by the yield strength (σ_y) at a strain value of 0.2%, and a simple empirical relation in the form of a power law called the Holloman equation which relates the hardening behavior to the plastic strain (ϵ_p) given by:

$$\sigma = K(\epsilon_p)^n$$

- K is the strengthening factor, a material constant.
- n is the strain hardening exponent.

Condition	σ_y (MPa)		K (MPa)		n	
	ED	CD	ED	CD	ED	CD
Area Reduction	85	117	279	279	0.10	0.10
SP- As worked	80	80	299	299	0.19	0.25
SP- Intermediate Anneal	87	80	262	255	0.22	0.15

- The yield strength of the Area Reduction tube is significantly different to the rest.
- From IPF in Fig.6(a) the texture along the circumference is mainly $\{111\}$ | CD.
- The $<111>$ orientations require activation of multiple slip for deformation, or higher yield strength [4].

Conclusions:

- Different uniform and consistent textures in the three sample conditions indicate redundant strain strategies are effective to provide microstructure options for seamless tubing for hydroforming.
- The relative strength, hardening, and elongation are sensitive to texture and grain level banding of the processed tube material

References:

1. W. Singer, Physica C: Superconductivity 441, 89–94 (2006).
2. S. Balachandran, R. Elwell, *et.al*, IEEE Transactions on Applied Superconductivity 23, 7100904–7100904 (2013).
3. V. Palmieri, R. Preciso, V. L. Ruzinov *et al.*, Nucl Instrum Meth A, vol. 342, no. 2-3, pp. 353-356, Mar. 1994.
4. K. Saito, T. Fujino, H. Inoue *et al.*, IEEE T Appl Supercon, vol. 9, no. 2, pp. 877-880, Jun. 1999.