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Enhancement of Flow Properties by Grain Refinement and Other Structural Modification

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Abstract

Recent techniques of severe plastic deformation including variants of rolling, equi-channel angular pressing and friction stir processing were used to enhance tensile properties of single phase, quasi-single phase and two phase alloys by grain refinement and substructural development. This led to the feasibility of mechanical processing of not only brittle material like hypereutectic Al-Si alloys, composites, and Mg alloys but also improved strengthening of ductile materials like Type 304 stainless steel. Presented here is a review of the recent works done in the author's group along with that emerged from collaboration elsewhere.

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Keywords: Severe plastic deformation, grain size, tensile properties, alloys, composites.

1. Introduction

Grain boundaries, in spite of being known as an imperfection in materials area, play important role in various fields including strengthening and ductilizing of most common category of materials called polycrystalline materials. Both grain boundary and grain interior of polycrystalline material contribute to workability and strengthening but their relative importance is sensitive to their proportions. For example, fine grained materials with significant proportion of grain boundaries below 10 µm grain size are stronger than coarse grained materials at low temperatures. The reverse is true at high temperatures. Approaching still finer grain size of nanocrystalline level dramatically changes the properties of materials, including lowering the high temperature type properties to even lower temperatures. On the other hand the materials above 100 µm grain size exhibit only marginal contribution of grain boundaries, owing to the dominance of grain interior, like seen as in improved creep resistance. Having stable fine grain size at elevated temperature exhibits superplasticity and this ease to deformation, *i.e.* minimum creep resistance of material, is exploited as a novel mechanical processing technique called superplastic forming. In view

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of this, some of the techniques of grain refinement in bulk material, called severe plastic deformation, are used here to study the nature of microstructure evolution and its effect on deformability in a range of materials, which include single phase, quasi-single phase, two-phase and composite materials.

Listed below are the various methods that constitute severe plastic deformation employed for grain refinement of bulk materials [1].

1. Accumulative roll-bonding (ARB)
2. High pressure torsion (HPT)
3. Severe plastic torsion straining (SPTS)
4. Repetitive corrugation and straightening (RCS)
5. Cyclic extrusion compression (CEC)
6. Torsion extrusion
7. Severe torsion straining (STS)
8. Cyclic closed-die forging (CCDF)
9. Super short multi-pass rolling (SSMR)
10. Equal channel angular pressing (ECAP)
11. Friction stir processing (FSP)

The methods used in the present work include rolling coupled with thermo-mechanical processing approach, rolling on the principle of repetitive corrugation and straightening (RCS), caliber rolling, equal channel angular pressing (ECAP) along with its variant, and friction stir processing.

2. Experimental Materials and Techniques

Listed in Table 1 are the materials investigated for grain refinement and evaluation of tensile properties.

Table 1. Material composition, processing technique, tensile test condition and grain size

S.N.	Chemical Composition (wt%)	Severe Plastic Deformation Technique	Grain size (µm)		Tensile/Compression Test Condition (all tensile tests unless stated otherwise)	Ref.
			Initial (d_0)/condition	Final Microstructure/ Grain Size (d_f)		
1.	Mg-4Li-1Ca	Thermo-mechanical processing using rolling	As-cast and homogenized coarse structure	3.5	Room temperature, strain rate 1×10^{-4} s^{-1}	[2]
2.	304 Stainless Steel: Fe-18.47 Cr, 8.10 Ni, 0.94 Mn, 0.53 Si, 0.066 C, 0.004 S, 0.039 P	Sine wave notch rolling (modified corrugated rolling) + plane rolling for flattening	45	No grain size change but numerous slip lines and shear bands	Room temperature, strain rate 1×10^{-4} s^{-1}	[3]
3.	AZ31B: Mg-3Al-1Zn-0.2Mn	Caliber rolling at elevated temperatures	33	3.6	Room temperature, strain rate 1×10^{-4} s^{-1}	[4]
4.	AA6082: Al-1Mg-1Si- 0.4Cu	Equi-channel angular pressing using L-shape die	Commercial grade	0.2	373 K to 623K; strain rate range: 10^{-4} to 10^{-1} s^{-1}	[5]
5.	Pb-Sn eutectic: 40.17 Pb, 0.016 Fe, 0.071 Cu, 0.041 Al, and balance Sn.	Equi-channel angular pressing using T-shape die	7.6: As-cast with dendrites, non-uniform distribution of phases	5.7	Room temperature, strain rate 5×10^{-4} s^{-1}	[6]
6.	Al- 34Si- 0.4Mg -0.3Fe - 0.3Cu	Friction stir processing	188 (Initial Si particle size)	1.3	------------	[7,8]
7.	A356 (Al-7Si-0.3Mg) + 5 B_4C composite	Friction stir processing	As-cast coarse microstructure	1.3	Differential strain rate compression tests: 10^{-3} to 10^{-1} s^{-1} and Temperature 743 and 773 K	[9]

Various standard techniques were used for metallography, tensile and compression tests, and microstructure examinations by electron microscopes, the details of which are presented elsewhere [3-9].

3. Results and Discussion

The various processing methods used for grain refinement and improving the tensile properties are listed in Table 1. The same are expanded below so as to appreciate the range of processes possible to obtain finer level of structures that give better properties than conventionally known in various materials. The references in the Table also correspond to the respective sections in the text to be described below.

3.1 Variants of Rolling

Three variants of rolling were used for grain refinement.

First, conventional flat rolling at two temperatures sequentially was used to produce fine grain size in Mg-4Li-1Ca alloy subsequent to homogenization [2]. The selection of temperatures and reduction per pass required was made to avoid cracking of work piece and formation of large grains. The microstructure obtained upon final rolling was found to be in order of submicrons, which could not be resolved at magnifications available in optical microscope. The initial microstructure having non-equiaxed grains changed into equiaxed grains of mean size 3.5 μm as seen after annealing of the rolled material at 623 K for 30 min. This led to significant increase in both tensile ductility and strength of Mg-4Li-1Ca alloy.

Second, repetitive rolling in the sequence of corrugation and straightening was performed [3] on solution annealed AISI 304 stainless steel coil of 1 mm thickness at room temperature by employing rolls of sine notch wave and flat contours. This was repeated to four cycles. Shown in Fig. 1 are the pictures of corrugated and then flat rolled coil upon completion of first cycle. As shown in Fig. 2, there appeared numerous shear bands and twins upon rolling with no change in grain size with increasing number of cycles. This led to increase in yield strength and tensile strength with increasing number of cycle up to predominantly first and second cycles with a corresponding decrease in ductility. Beyond second cycle, the change in tensile properties appeared very marginal or negligibly low as illustrated in Fig. 2 for yield strength and ductility plotted as a function of number of cycles. Interestingly, it is seen to have ductility of better than 40% although the yield strength has increased four folds from the initial value of 200 MPa to about 800 MPa after four cycles.

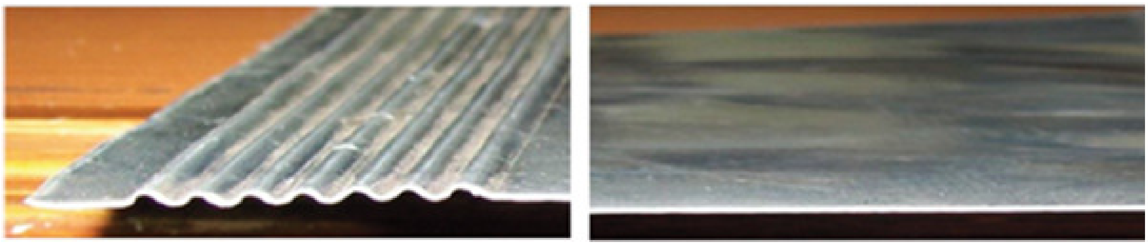


Fig. 1. Example of a coil undergone one cycle of sine notch wave rolling followed by flat rolling [3].

Third, caliber rolling, which consists of a sequence of grooves of decreasing dimensions in the rolls as shown schematically in Fig. 3(a), was used to roll a rod of 12 mm diameter from mill plate AZ31B: Mg-3Al-1Zn-0.2Mn alloy of thickness 50 mm at selected elevated temperatures of 523, 573, 623, 673 and 723 K [4]. The grain sizes obtained after caliber rolling at different temperatures were much smaller than the initial grain size but the final grain size did not follow any systematic effect of caliber rolling temperature. Shown in Fig. 3(b, c) are the initial and final microstructures after caliber rolling at 573 °C, with reduction in grain size from initial value of 33 μm to 3.6 μm.

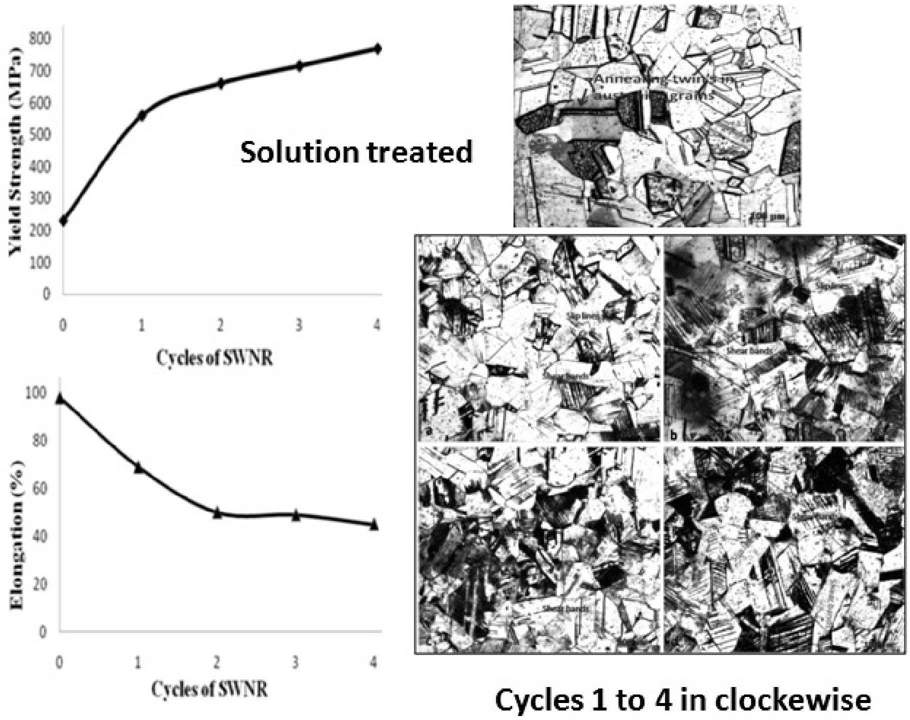
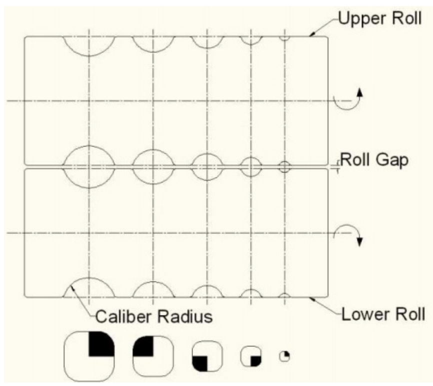


Fig. 2. Microstructures of initial coil (solution treated) and upon 1, 2, 3, and 4 cycles of sine notch wave rolling (in clockwise direction from top left corner) and their effects on yield strength and ductility [3].



(a)

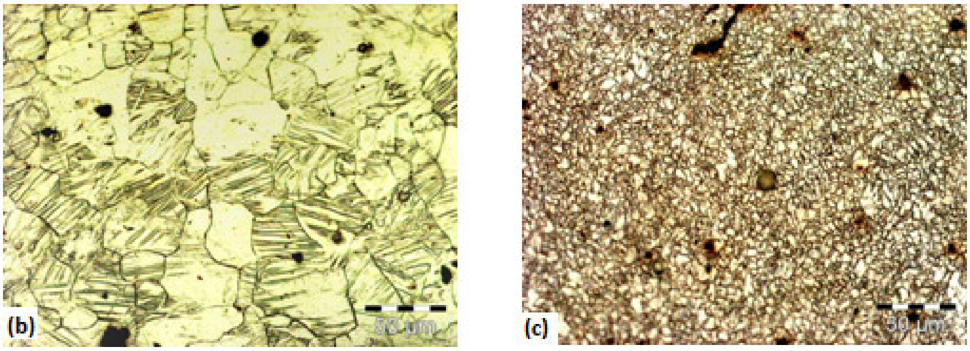


Fig. 3. (a) Schematic of caliber rolling; optical micrographs of AZ31B Mg-alloy: (b) mill rolled plate, (c) Caliber rolled at 573 K [4].

Not only that the grain size varied irregularly with caliber rolling temperature but also no systematic effect of caliber rolling on tensile properties at room temperature was noticed. In fact, the yield and tensile strength plotted (not shown here) as a function of grain size, obtained from different caliber rolling temperatures, also did not follow Hall-Petch type relationship. The irregular effects of caliber rolling on grain size and tensile properties, *viz.* tensile strength and ductility, are presented in Fig. 5. The tensile strength obtained here (290 MPa) is greater than the earlier reported strength values (256 MPa) for this material, while the ductility remained similar (22% here *vs* 22.2% earlier) [10]. The reason for irregular effect of caliber rolling temperature may be due to the difference in the twin density and texture both being influenced by rolling temperature.

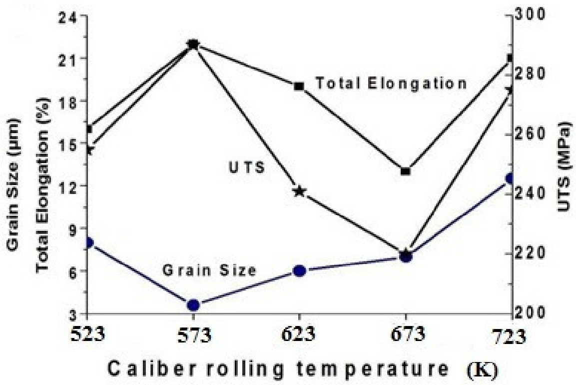


Fig. 4. Plot of tensile properties and grain size in AZ31B Mg-alloy as a function of caliber rolling temperature over 523 K to 723 K [4].

3.2. Equi-Channel Angular Pressing

Conventional L-shape equi-channel angular pressing was used for grain refinement of a commercial grade AA6082 Al-alloy: Al-1Mg-1Si- 0.4Cu [5]. Billets with the dimensions $100 \times 10 \times 10$ mm^3 were cut for ECAP in a die with the internal channel angle $\varphi = 90°$ and outer angle $\psi = 0°$. Each sample was subjected to eight ECAP passes with a rotation by 90° about the pressing direction after each pass (route Bc). The strain per pass is calculated according to eq. (1). The strain produced in each pass was about 1, so that the cumulative strain that the specimen underwent as a result of ECAP processing was about 8. The temperature during ECAP was maintained at 373 K.

$$\varepsilon = \frac{1}{\sqrt{3}}\left\{2\cot\left(\frac{\phi}{2}+\frac{\psi}{2}\right)+\psi\cos ec\left(\frac{\phi}{2}+\frac{\psi}{2}\right)\right\} \quad \text{Eq. (1)}$$

The TEM examination revealed the formation of a refined microstructure that consisted of grains and subgrains having a mixture of high-angle and low-angle boundaries (Fig. 5), which is a typical of SPD-processed metals and alloys. The SAD pattern reveals evidence of recrystallized grains with some regions still remaining heavily distorted due to severe plastic deformation. The There were three types of grains observed in the microstructure: (1) grains free from dislocations in their interior, (2) grains with low dislocation density in the interior, and (3) grains with high dislocation density. The grain size varied in the range of 0.2 to 0.4 µm. A few very fine grains free of dislocations with an average size of 90 nm were also observed.

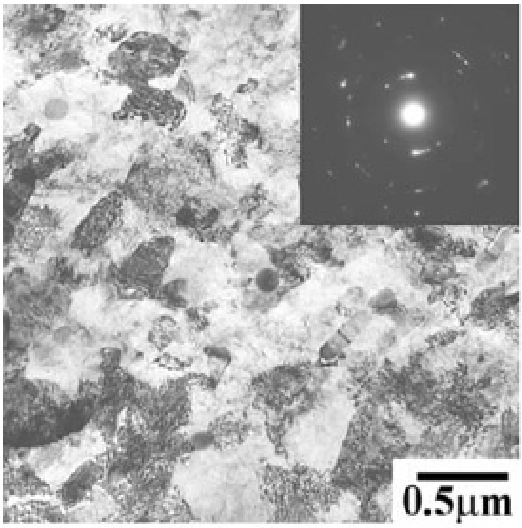


Fig. 5. TEMicrograph showing the structure evolved by equi-channel angular pressing of AA6082 Al-alloy: Al-1Mg-1Si- 0.4Cu to eight passes at 373 K; the SAD pattern reveals evidence of recrystallized grains with some regions still remaining heavily distorted [5].

Tensile tests were done at constant temperatures over the range 373 K to 623 K and at constant strain rates in the range 10^{-4} to 10^{-1} s^{-1}. Shown in Fig. 6 are the stress-strain curves obtained at various test temperatures at the strain rate of 1×10^{-3} s^{-1}. As expected, the flow stress decreases with increasing temperature. Similarly, deformation at constant temperatures but at varying constant strain rates exhibited decrease in flow stress with the decrease in strain rate. The strain rate sensitivity between strain rates 2.2×10^{-4} and 10^{-3} s^{-1} was determined from differential strain rate test to be 0.17. This suggests the deformation to take place by dislocation climb creep mechanism.

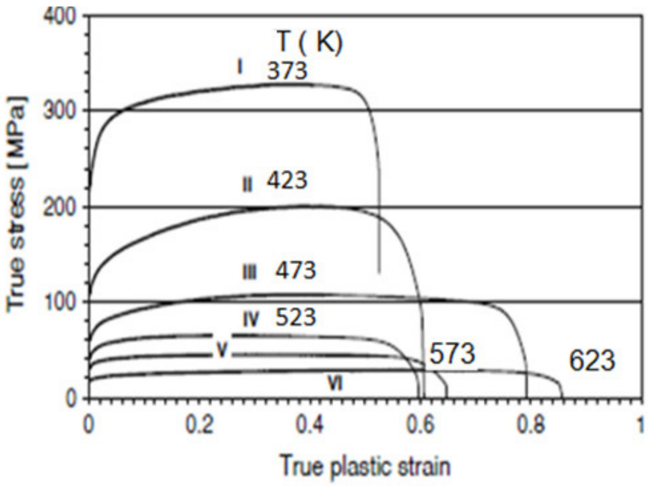


Fig. 6. Stress-strain plot for AA6082 Al-alloy: Al-1Mg-1Si-0.4Cu subjected to eight passes ECAP at 373 K, and tensile tested at various test temperatures (labelled along the curves) and strain rate 1×10^{-3} s^{-1} [5].

A 12 mm × 12 mm × 100 mm Pb-Sn eutectic of composition (wt%) Sn- 40.17Pb-0.016 Fe-0.071 Cu-0.041Al was subjected to equi-channnel angular pressing up to four passes at room temperature by employing a T-shape die [6]. The arrangement of the rod in the die is shown in Fig. 7(a) along with the microstructure in Fig. 7(b) that resulted upon four passes of ECAP. The initial as-cast structure was noted to be broken and fine equi-axed grains of size 5.7 μm with homogeneous distributions of the two phases in this alloy was obtained.

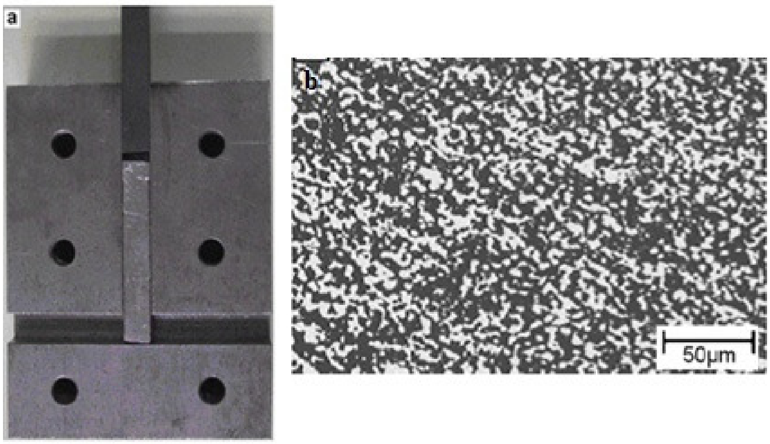


Fig. 7. (a) The arrangement of Sn- 40.17Pb- 0.016Fe- 0.071Cu- 0.041Al rod in the die along with the microstructure in (b) that resulted upon four passes of ECAP [6].

Tensile tests performed at room temperature and strain rate of 5×10^{-4} s^{-1} revealed progressive softening and enhancement in ductility from as-cast stage up to four passes as shown in Fig. 8. The maximum ductility of 230% was obtained upon four passes, which is less than the ductility values known from earlier works on this material, but this could be increased further by increasing the number of ECAP passes. However, under the condition used for ECAP, further passes could not be carried out successfully as the rod developed some cracking.

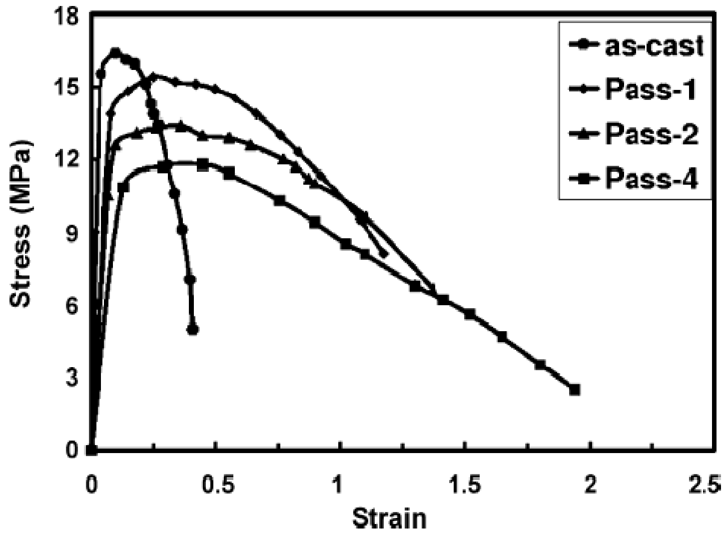


Fig. 8. Stress-strain curves showing the effect of number of ECAP passes in Sn-40.17Pb-0.016Fe-0.071Cu- 0.041Al alloy up to four passes of ECAP at room temperature and strain rate of 5×10^{-4} s^{-1} [6].

3.3. Friction Stir Processing

An Al-alloy permanent mould cast plate of dimensions 130 mm × 70 mm × 12 mm of composition (wt%) Al–30Si–0.2Mg–0.1Fe was subjected to friction stir processing [7]. Single pass and two pass (with 100% overlap over the first pass FSP layer) FSP experiments were performed using conventional milling machine set-up and a tool made of H13steel having flat shoulder diameter of 25 mm and pin of 6 mm diameter. Throughout the experiment the tool rotation and tool translation speeds were kept constant at 1000 rpm and 16 mm/min respectively. Further work [8] on FSP was done on larger plate using other machine with different tool dimension and FSP condition. Shown in Fig. 9 [7, 8] are the micrographs of as-cast (see Fig. (b)) and two pass friction stir processed (see Fig. (c)) samples with all the zones (a) consisting of stir zone, base metal and thermo-mechanical affected zone between them (not marked). As listed in Table 1, it is encouraging to find that massive large Si particles of 188 µm are broken into very small particles of size of 1.3 µm, the matrix grains are refined and the pore density is dramatically reduced.

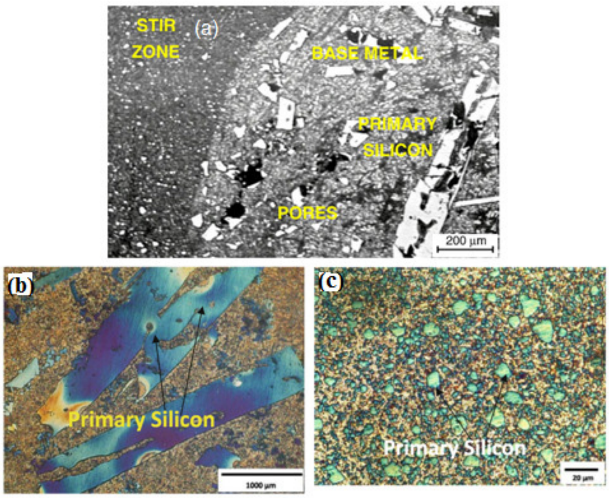


Fig. 9. Micrographs of as-cast (b) and two pass friction stir processed (c) Al–30Si–0.2Mg–0.1Fe samples with all the zones (a) consisting of stir zone, base metal and thermo-mechanical affected zone between them (not marked) [7,8].

Friction stir processing was also attempted on an A356 (Al-7Si-0.3Mg) + 5% B_4C composite [9]. The grain refinement upon first pass FSP exhibited superplastic behavior with m = 0.44 in the lower strain rate regime of 1×10^{-3} to 5×10^{-2} s^{-1} at test temperature of 773 K in compression. At higher strain rates and lower test temperature, no characteristic of superplasticity could be revealed in spite some more grain refinement during further pass of FSP.

4. Summary and Conclusions

Severe plastic deformation processes like equi-channel angular processing and friction stir processing are found to significantly refine the microstructure, but the effect of the latter appears dramatic even just after first pass. FSP used for hard-to-work material like hypereutectic Al-34Si alloy results in reduction of Si particle size from initial value of 188 µm to 2.5 µm, and A356 (Al-7Si-0.3Mg) + 5% B_4C composite can be refined to exhibit superplasticity. However, rolling process, when extended with special roll design as for repetitive corrugation and straightening (RCS) process or caliber rolling, can produce a large quantity of fine grained or workhardened material easily. This is illustrated for AZ31B Mg-alloy and 304 stainless steel, respectively. However, structure-property relationship based on conventional effect of grain size on strength needs more structural parameters to understand flow properties. Conventional rolling process with suitable thermo-mechanical steps can be promising as established in seventies and eighties in the last century. It would be interesting to extend the more recent severe plastic deformation to thermo-mechanical treatment in order to explore the structure and property beyond known today.

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