Fabrication of Bulk Nanocrystalline and Ultrafine Grain Materials by Friction Stir Processing

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Outline of Presentation

- Background
- Objective
- Experimental
- Results
- Discussion
- Summary

Background I

Current processing methods for nanocrystalline metals and alloys:

- **Powder metallurgy**
  - **Inert gas condensation and consolidation**
  - **Mechanical alloying**
- **Crystallization of amorphous material**
- **Deposition methods**
  - **Electrodeposition**
  - **Physical vapor deposition**

Advantages	**Disadvantages**
Can produce materials with grain sizes below 100 nm	**Difficult to create full-density, porosity-free bulk materials**

Background II

Severe plastic deformation (SPD) techniques:

- Equal-channel angular pressing or extrusion (ECAP/E)
- High-pressure torsion straining
- Accumulative roll-bonding (ARB)

Achievements

Produce nanostructure bulk materials from ductile metals and alloys of low to moderate initial strength

Problems

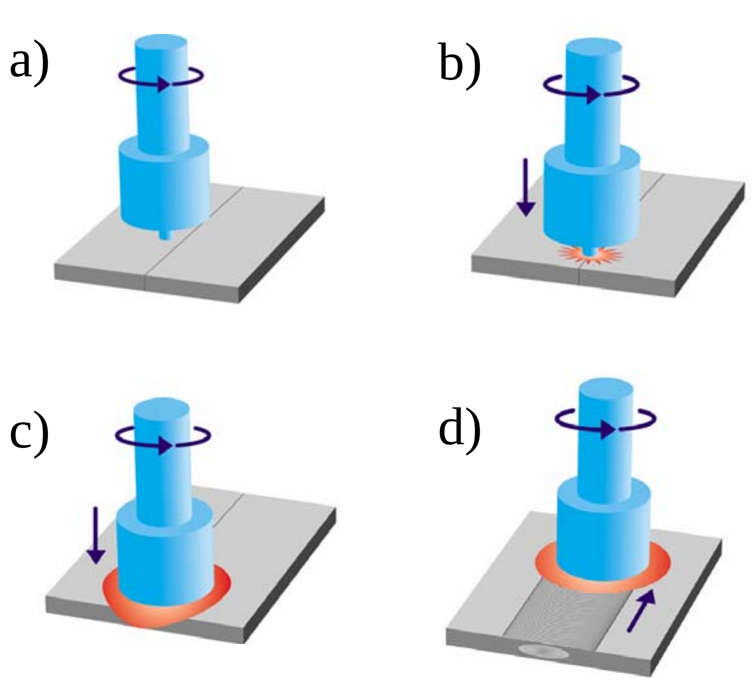
- Difficult to process high-strength metals and alloys
- Difficult to scale up

So far, established techniques can not produce full-density nanomaterial in bulk forms large enough for most engineering applications. Development of processing techniques which enable the production of bulk nanostructured materials large enough for many engineering applications is essential.

Background III

Friction stir processing (FSP) technique:

- Variation of friction stir welding (FSW)
- Thermo-mechanical processing
- Very large strain and high strain rate



Schematic courtesy of Rockwell Scientific

Process:

- Plunge a rotating tool into the material
- Localized heating produces
- Translate rotating tool

FSP is an effective grain refinement technique.
7075 Al: 3~4 μm

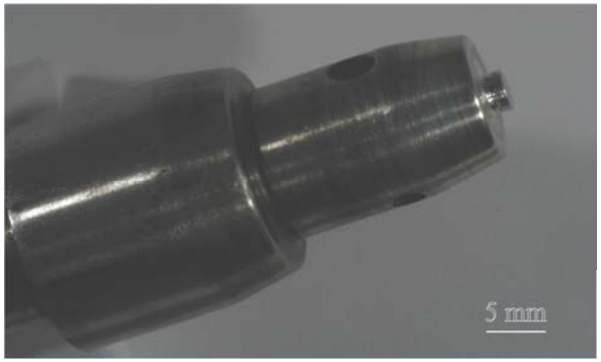
Objective

Use a new thermo-mechanical processing technique, friction stir processing (FSP), combined with rapid cooling, to produce large scale fully dense nanocrystalline and ultra-fine grain materials.

Experimental

Tool dimensions:

- Shoulder diameter: 9 mm
- Pin length: 1.9 mm
- Pin diameter: 3 mm



Tool used in this study

Processing parameters:

- Tool rotation: 800 rpm
- Travel speed: 10 cm/min
- Cooling liquid: mixture of water, methanol and dry ice.

Material:

- Commercial 7075 Al
- 2 mm sheet

Microstructure observation:

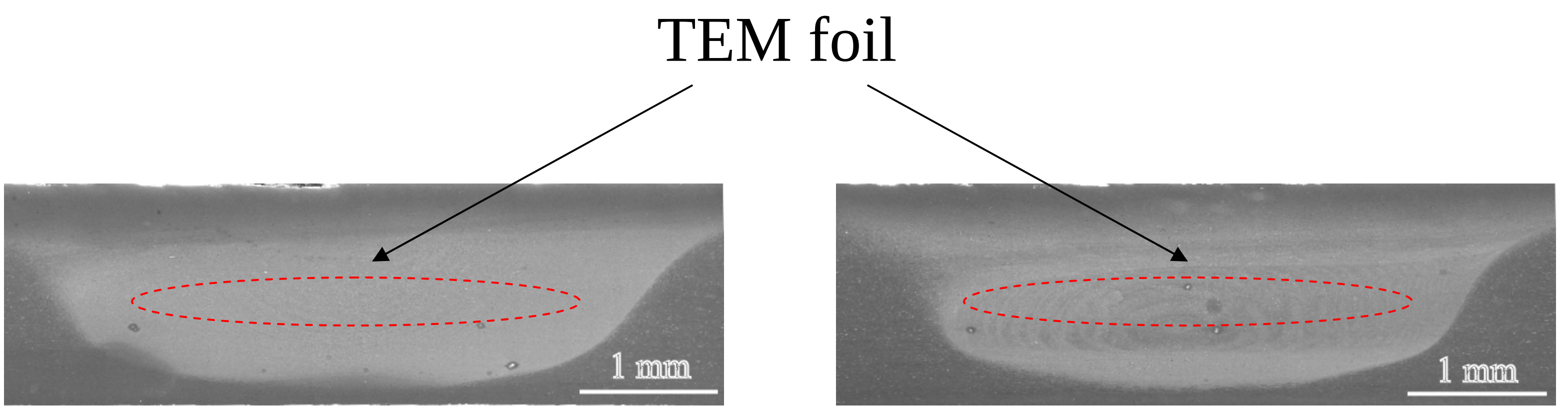
- TEM

Cooling rate was controlled by adjusting the volumetric flow rate of cooling liquid. A greater flow rate of cooling fluid caused faster cooling.

Processed Material

Specimen A
(faster cooling)

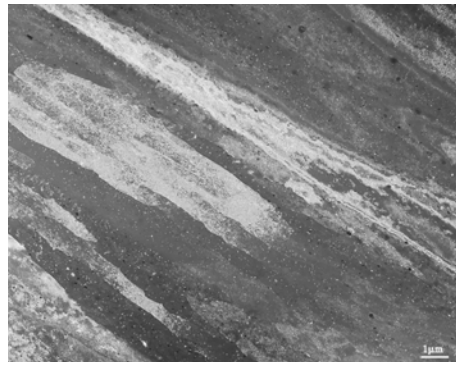
Specimen B
(slower cooling)



Cross section of processed material

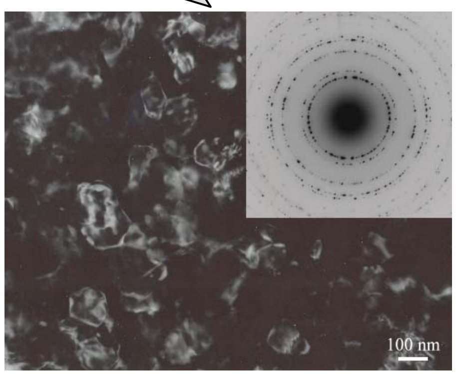
Microstructure Characterization

Grain size:
30-150 nm (~100 nm)
Ring diffraction:
many small grains with random misorientations

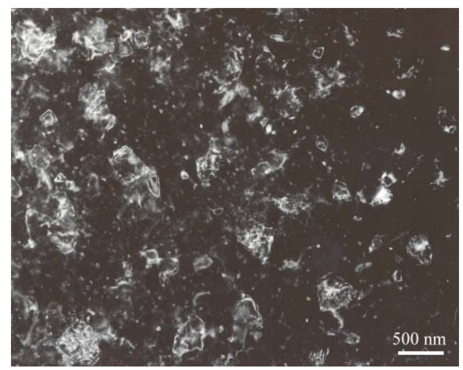


Parent material

Grain size:
100-400 nm
Average:
~300 nm



Specimen A

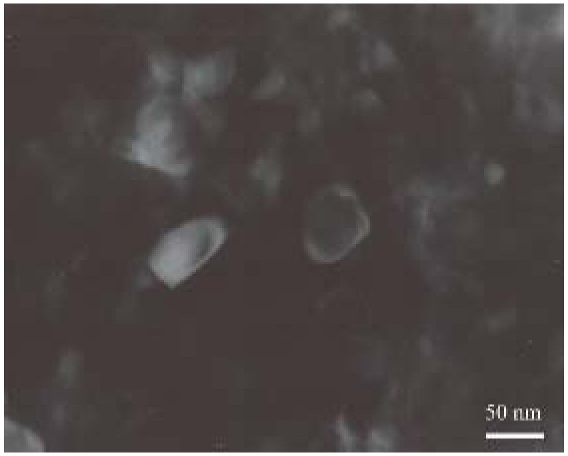


Specimen B

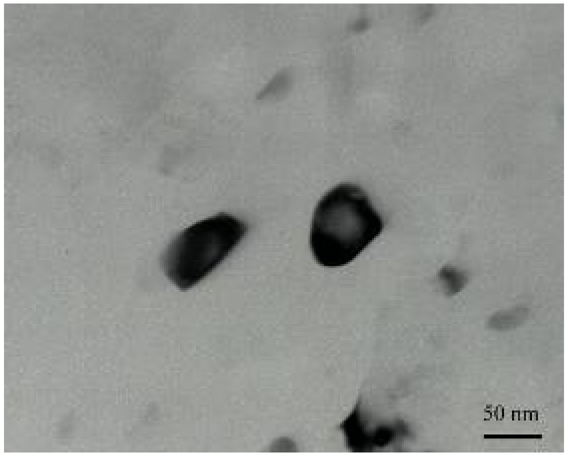
Nanocrystalline structure was produced in a single pass during FSP.
Obtained grain size can be controlled by changing cooling rate.

Extremely Fine Grains

Specimen A



Bright-field image

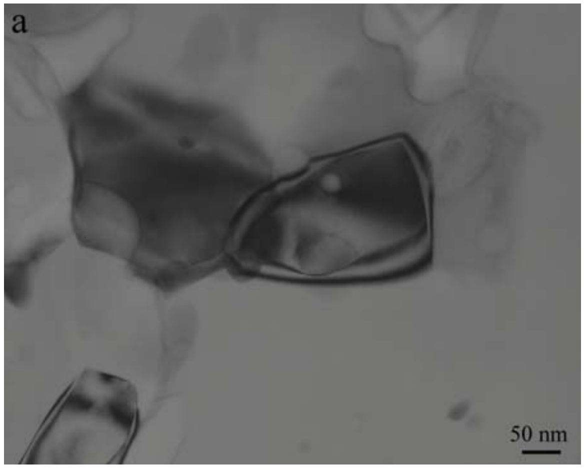


Dark-field image

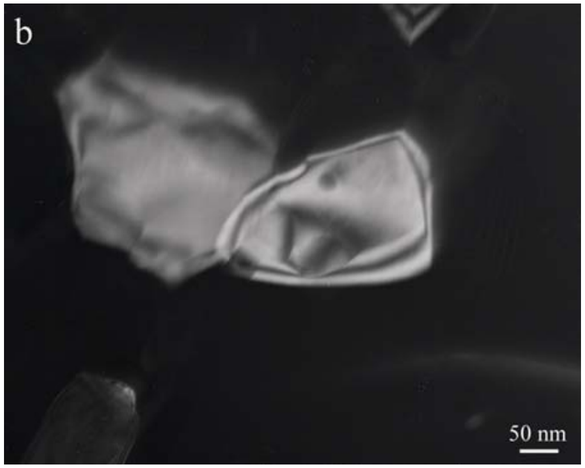
Large difference in diffraction contrast with the neighboring grains in the dark field image indicates that these are not sub-grains, but separated by high-angle boundaries.

Larger Grains

Specimen A



Bright-field image



Dark-field image

- **Dislocation free**
- **Specific "diffusive" diffraction contrast of inclined grain boundaries:**
  - i) **Non-equilibrium GBs with higher energy.**
  - ii) **Long range stresses caused by an absorption of large number of lattice dislocations.**

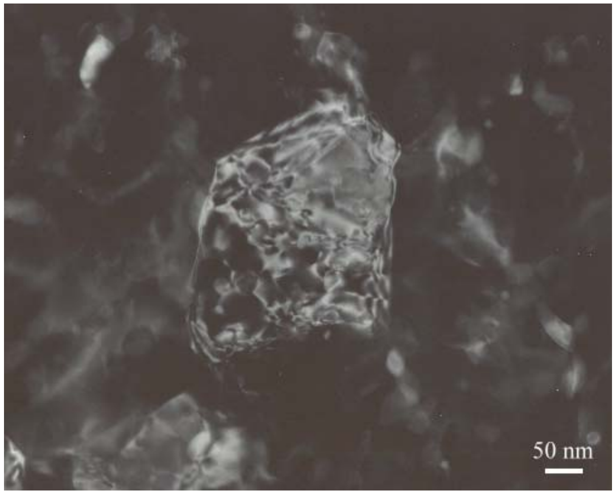
Microstructure in Sample B

Small grains (< 200 nm)

- Free of dislocations
- Non-equilibrium boundaries

Larger grains

- Dislocations



Dislocation structure in larger grain

Discussion

▎Dynamic Recrystallization (DRX) Mechanism

Aluminum

- High stacking fault energy
- Dynamic recovery

DRX in aluminum

- High purity Al: very high mobility of grain boundaries
- AlMgMn: large particles stimulate nucleation

7075 Al

Dynamic recovery is principal restoration mechanism

Discussion

Friction Stir Processing
Nano-scale grains were created

⇩

Microstructure Characterization
Nanocrystalline structures consist of high-angle boundaries. Even the very small grains (~ 30 nm) were found to be separated by high-angle boundaries, instead of being sub-grains.

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Discontinuous Dynamic Recrystallization

During FSP, a complex stress state and strain components with very large strain gradients were caused in the processed material. Furthermore, large amounts of dislocations were introduced to accommodate the strain incompatibility.The complex stress state, complicated strain patterns and dislocation configurations, and high density of dislocations are all beneficial in allowing copious nucleation during DRX.

Discussion

Ⅱ Microstructure Evolution During FSP

In the early stage of the process, a large number of nuclei are formed in deformed microstructure

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During the subsequent thermal-mechanical deformation, grains grow absorbing many dislocations to form non-equilibrium GBs

⇩

Additional dislocations are generated by further plastic deformation within the larger grains which accommodate strains preferentially.

Final microstructures are strongly dependent on the processing parameters and cooling rate

Summary

FSP is a fairly effective technique in refining grain sizes to the nanometer or sub-micrometer level. In summary, FSP has several advantages over other nano processing methods which include:

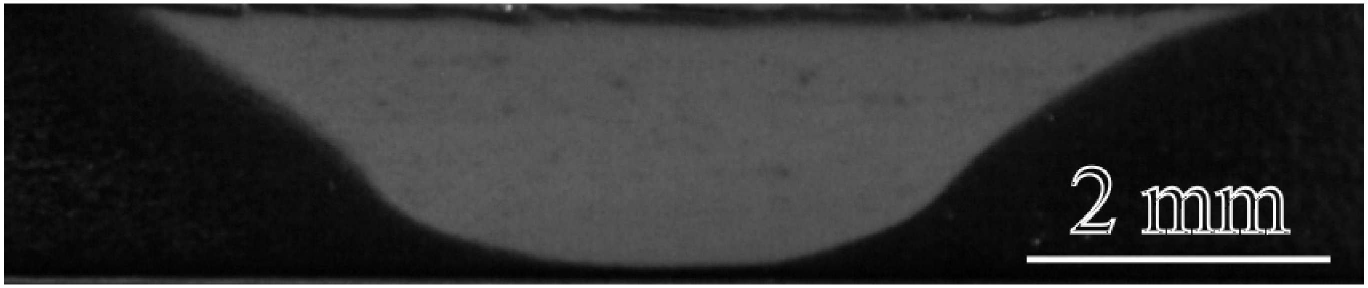
i) ability to produce nano-grains with high angle boundaries in a single step.

ii) ability to to control the resulting microstructures by changing the processing parameters and cooling rate.

iii) potential to process an entire sheet via multi-pass overlapping sequence to obtain a desired microstructure over a fully dense large area.

Multi-pass Overlapping Processing Technique

Single-pass processing



Four-passes processing