Friction Stir Reaction Processing to Develop Sphene on Aluminum Substrate

Research Undergraduate: Michelle Loomis
Faculty Advisors: Mr. William Arbegast, Dr Michael West
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SOUTH DAKOTA SCHOOL OF MINES & TECHNOLOGY

Objectives

- Create sphene ($CaTiO(SiO_4)$) by Friction Stir Reaction Processing (FSRP)
- Use Scanning Electron Microscopy (SEM) and X-ray diffraction to determine if compounds formed

Materials

- Calcium Oxide (CaO), Calcium Carbonate ($CaCO_3$), Silica (SiO_2), Titania (TiO_2) powders
- Aluminum 1100 plates, Aluminum 3011 1/8 inch square tube, Aluminum foil tape

Procedure

- Use ThermoCalc to determine thermodynamically favorable powder combinations
- Thoroughly mix powders by ball milling in methanol overnight
- Pack powder into tube and set up as shown in figure 1
- Friction stir weld at 1400 revolutions per minute at a speed of 1 inch per minute
- Cut welds in the longitude and transverse directions, prepare metallographic samples
- Analyze samples by SEM and XRD

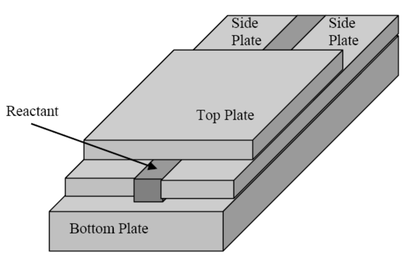

Figure 1 Aluminum plates with reactant embedded for FSRP [1]

Results & Discussion

Optical Microscopy

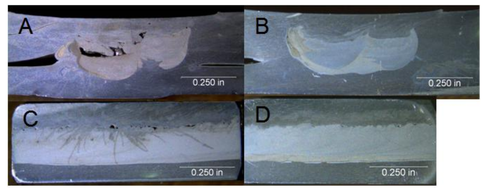

Figure 2 shows the cut , polished and etched welds in the transverse and longitude direction. Left: CaO welds, Right: $CaCO_3$ welds. The lighter color gray area is the nugget zone, which is where the powders are stirred into.

Scanning Electron Microscopy

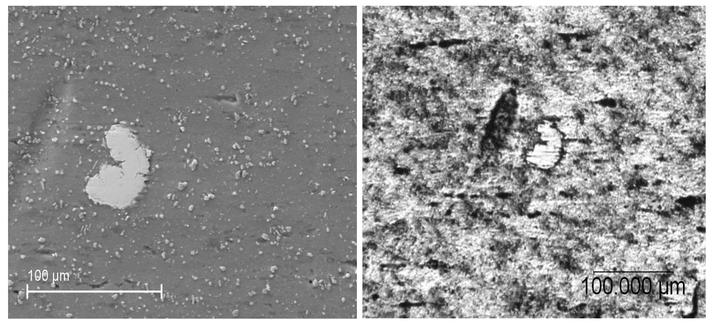

Figure 3 Left: SEM image of large particle at 1000X Right: Optical microscope image of particle at 800X

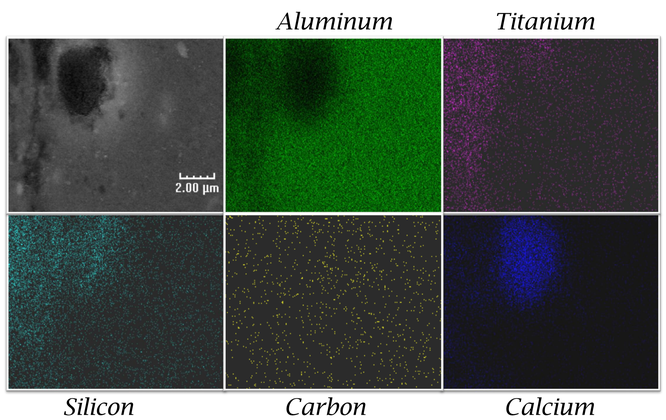

Figure 4 X-ray map image of particles in stir zone

X-Ray Diffraction

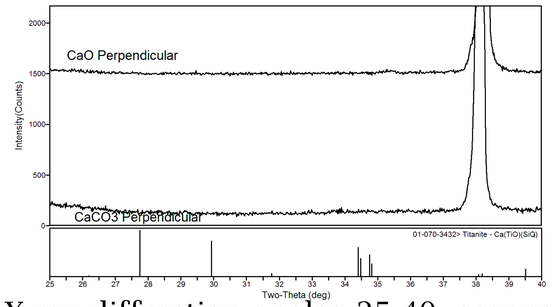

Figure 5 X-ray diffraction angles 25-40, scanned at a speed of 0.5 degrees per minute. Shows no evidence of sphene peaks.

Conclusions

- Powder was well distributed throughout the weld nugget
- Particles were too fine to resolve using SEM
- Particles were either too fine, or not in enough concentration to detect through XRD

Future Work

- Use TEM to resolve fine particles and determine compounds
- Reproduce experiment with different weld parameters to increase temperature.

Acknowledgements

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References

- [1] Howard, S.M., Jasthi, B.K., Arbegast, W.J., Grant, G.J. & Herling, D.R.(2005). *Friction stir reaction processing in aluminum substrates.* The minerals, metals & materials society