



HUMAN CAPITAL
NATIONAL COHESION STRATEGY



INSTITUTE OF METALLURGY
AND MATERIALS SCIENCE
Polish Academy of Sciences

EUROPEAN
UNION



Aluminium matrix nano-composites reinforced with aluminium nitrides

PhD student: Marta Gajewska

Supervisor: Prof. Jerzy Morgiel

• Interdisciplinary PhD Studies in Materials Engineering with English as the language of instruction •

Institute of Metallurgy and Materials Science

Polish Academy of Sciences

Reymonta 25, 30-059 Krakow, tel. +48 12 295 28 00, fax +48 12 295 28 04

www.imim-phd.edu.pl

Project is co-financed by European Union within European Social Fund



Research outline

Ist year

IInd year

1. Literature review:

- selection of materials (matrix/reinforcement)
- selection of available production method

2. Preliminary comparative study:

- AlN vs. Al₂O₃ reinforcement
- matrix vs. composite

3. Equipment trainings:

- X-ray diffractometry training on a two-stand Philips X-ray PW 1710 and PW 1830 diffractometer with X'Pert system
- SEM training on FEI XL30 E-SEM
- training on CSM-Instruments tester concerning Vickers microhardness measurements
- TEM training on Tecnai G2 F20
- training in Ar ion milling with Leica EM RES101

4. Training in preparation of samples for OM/SEM/TEM investigations

5. Optimization of PM production route:

- high energy ball milling
- hot pressing

6. Composite production:

- Al/AA7475 matrix
- AlN reinforcement of different particle size: <40 μm (-325 mesh), ~1 μm, <1 μm
- amount of reinforcement: 5, 10, 20 wt.%

7. Materials characterization:

- investigation of ball milled composite powders (XRD, OM, SEM, microhardness tests)
- investigation of composite compacts (OM, SEM, TEM, hardness, microhardness, compressive strength tests)

8. Attempt to elaborate an *in situ* method for aluminium/AlN composite production

(via reactive ball milling)



Materials

Composites

7475 aluminium alloy powder
(wt.%,: 5.7 Zn, 2.2 Mg, 0.7 Fe, 1.6 Cu, 0.1 Mn, 0.5 Zr, rest Al)



matrix

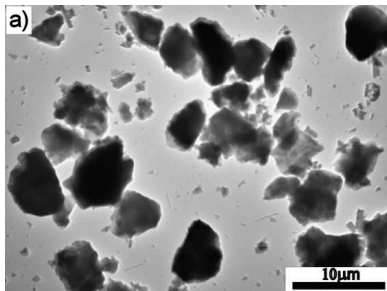


Aluminium nitride powders:

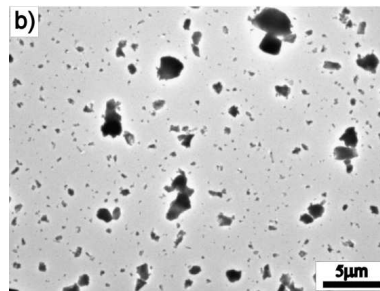


reinforcement

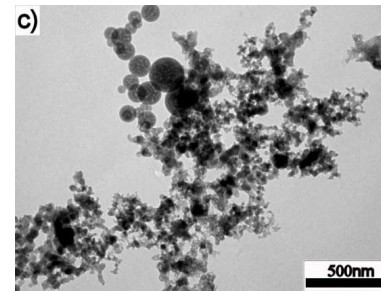
a) <math>< 40 \mu\text{m}</math> (-325 mesh)



b) $\sim 1 \mu\text{m}$



c) $< 1 \mu\text{m}$



Reference materials

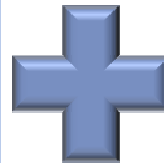
7475 aluminium alloy with/without Al_2O_3 reinforcement
Al (99,99%) with/without AlN reinforcement



Composite production route

High energy ball milling:

- 5, 10, 20% of ceramic phase
- milling time (40h)
- atmosphere (Ar)
- stearic acid (PCA - process control agent)
- bearing steel balls
- tool steel containers
- 10:1 balls to powder ratio
- rotational speed: 200 rpm



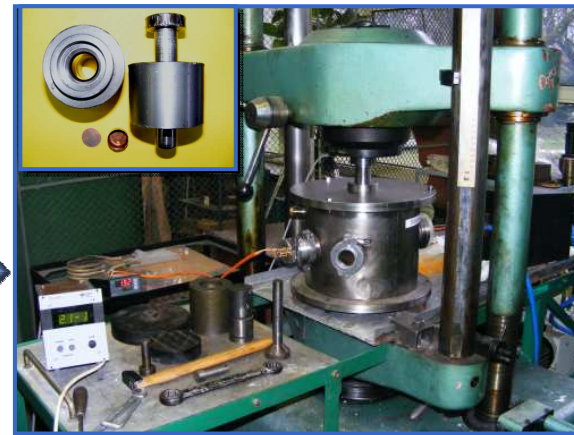
Hot pressing:

- temperature (380°C)
- holding time (10 min.)
- vacuum ($\sim 10^{-2}$ bar)
- pressure of 600 MPa
- copper container
- steel mould



Planetary Fritsch
mill
Pulverisette 5

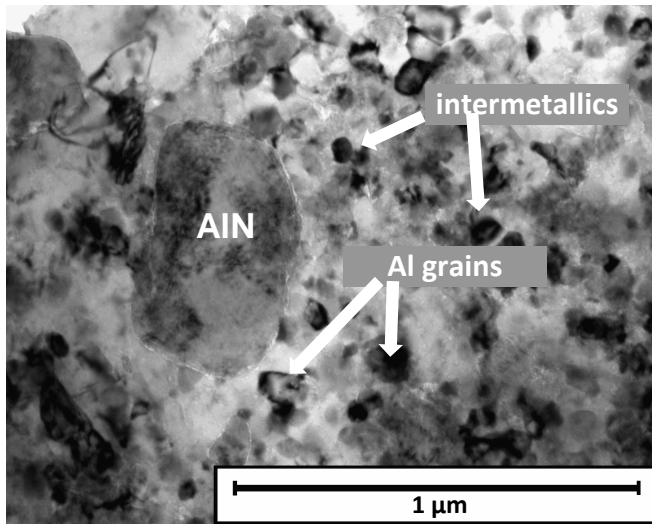
VEB 40 hydraulic uniaxial
press heated by high
frequency generator



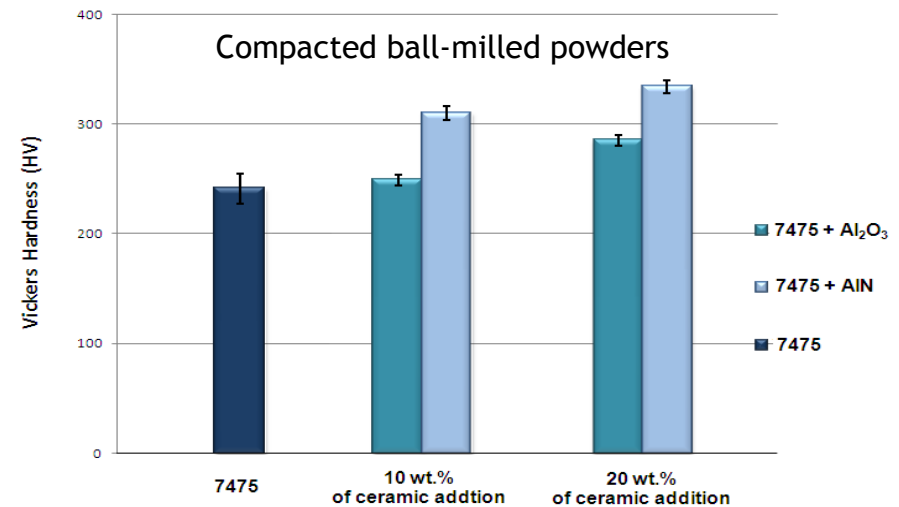


Preliminary investigations

Microstructure of ball milled/hot pressed
AA7475/ $\text{AlN}_{10\%}$



AlN vs Al_2O_3 reinforcement



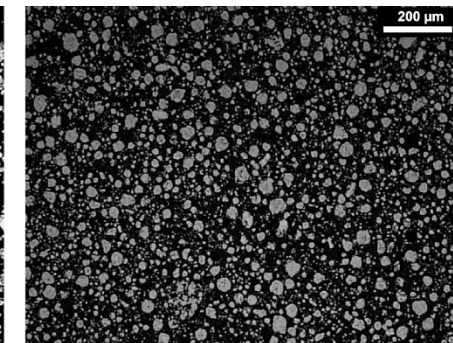
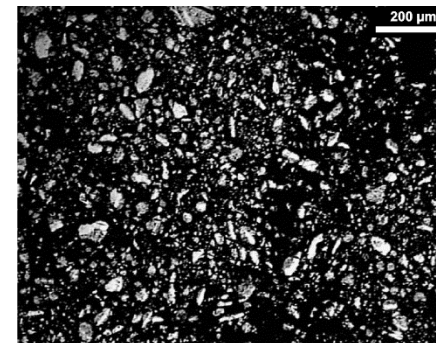
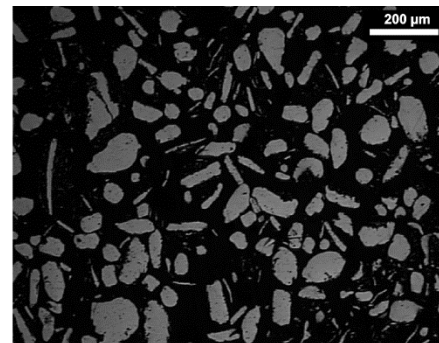
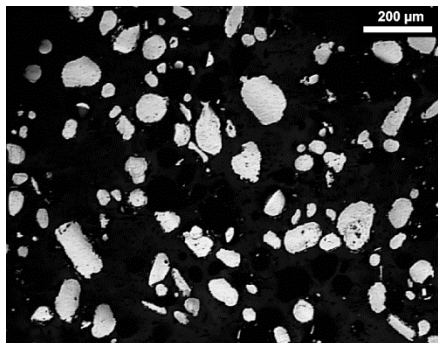
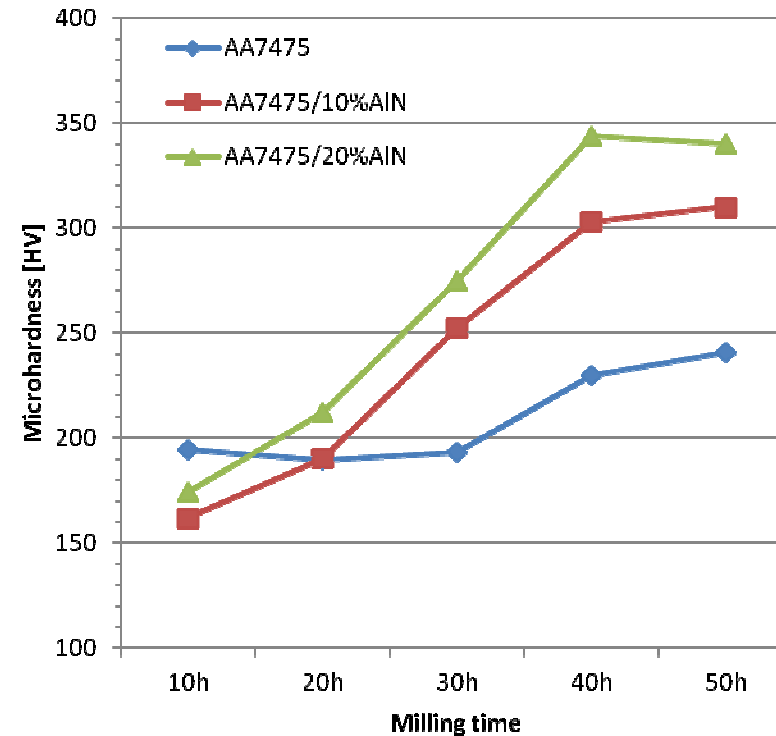
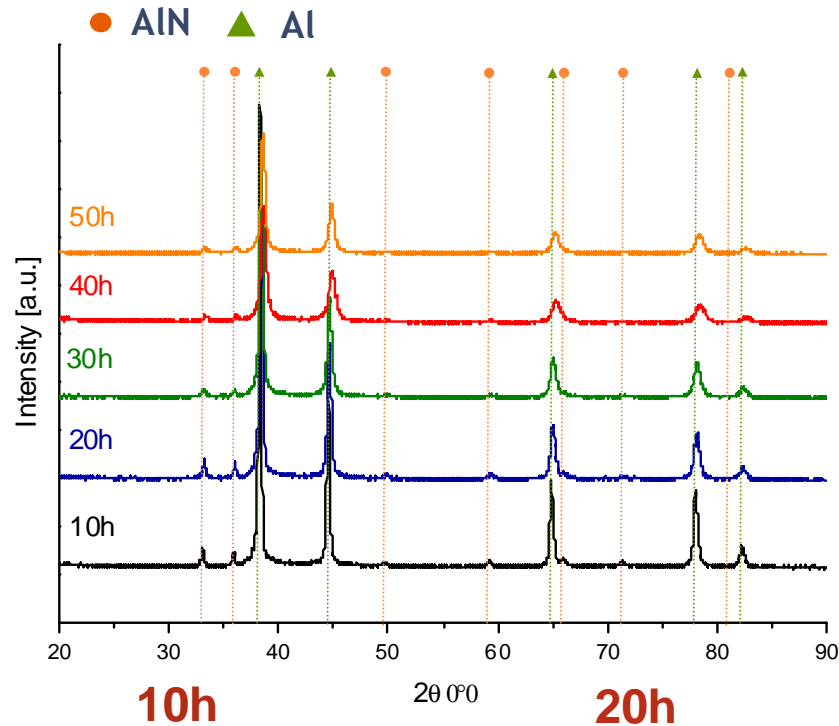
- hot pressing of ball-milled 7475 aluminum alloy powder with AlN particles allowed to obtain composites of a nanocrystalline matrix
- composites showed a **good dispersion of ceramic phases**
- higher strengthening effect over the matrix was achieved using AlN reinforcement

„Microstructure and properties of 7475 aluminum alloy matrix nano-composites with 10-20% of Al_2O_3 or AlN additions”,
M. Gajewska, J. Dutkiewicz, L. Lityńska-Dobrzyńska, J. Morgiel, Kompozyty 11:2 (2011) 142-146



Composite powder characteristics

Example: Ball milled AA7475 with 10% of $\sim 1\mu\text{m}$ AlN





HUMAN CAPITAL
NATIONAL COHESION STRATEGY



INSTITUTE OF METALLURGY
AND MATERIALS SCIENCE
Polish Academy of Sciences

EUROPEAN
UNION

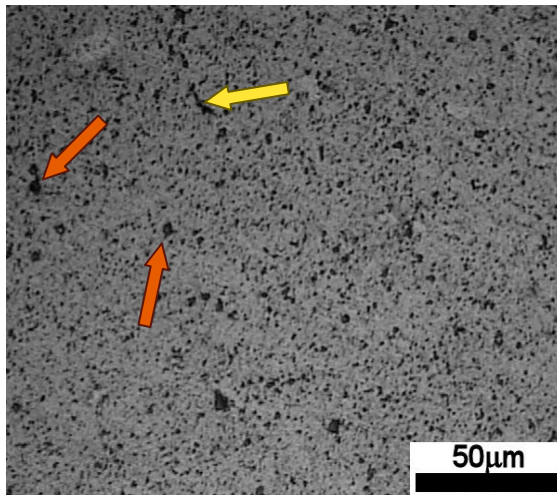


Composite characterization (1):

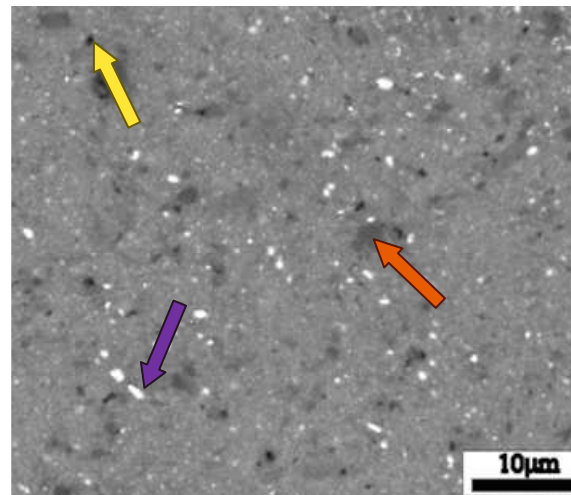
Microstructure

Example: ball milled/hot pressed AA7475 with 20 wt.% of $\sim 1\mu\text{m}$ AlN

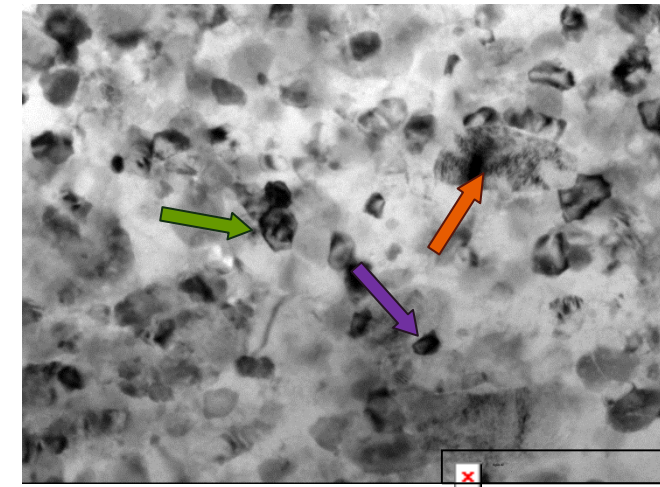
OM



SEM (BSE)



TEM (BF)



→ AlN

→ voids

→ intermetallics

→ Al grains

“Microstructure and mechanical properties of AA7475/AlN compacts with varied reinforcing particles size”, M. Gajewska, J. Dutkiewicz, J. Morgiel, accepted for publication in COMPOSITES THEORY AND PRACTICE

„Effect of reinforcement particle size on microstructure and mechanical behavior of AlZnMgCu/AlN nano-composites produced using mechanical alloying” submitted to Journal of Alloys and Compounds



HUMAN CAPITAL
NATIONAL COHESION STRATEGY



INSTITUTE OF METALLURGY
AND MATERIALS SCIENCE
Polish Academy of Sciences

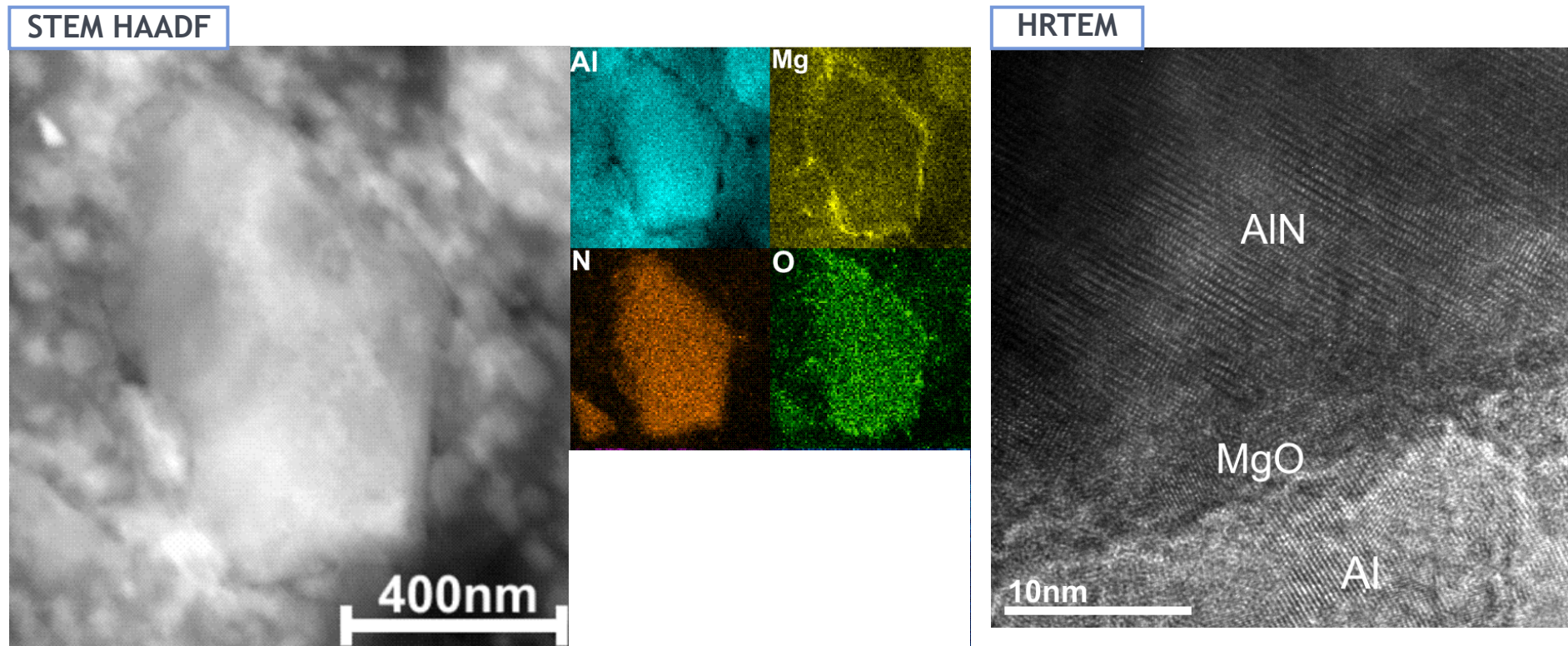
EUROPEAN
UNION



Composite characterization (2):

Metal/ceramic interface investigation

Example: ball milled/hot pressed AA7475 with 20 wt.% of AlN

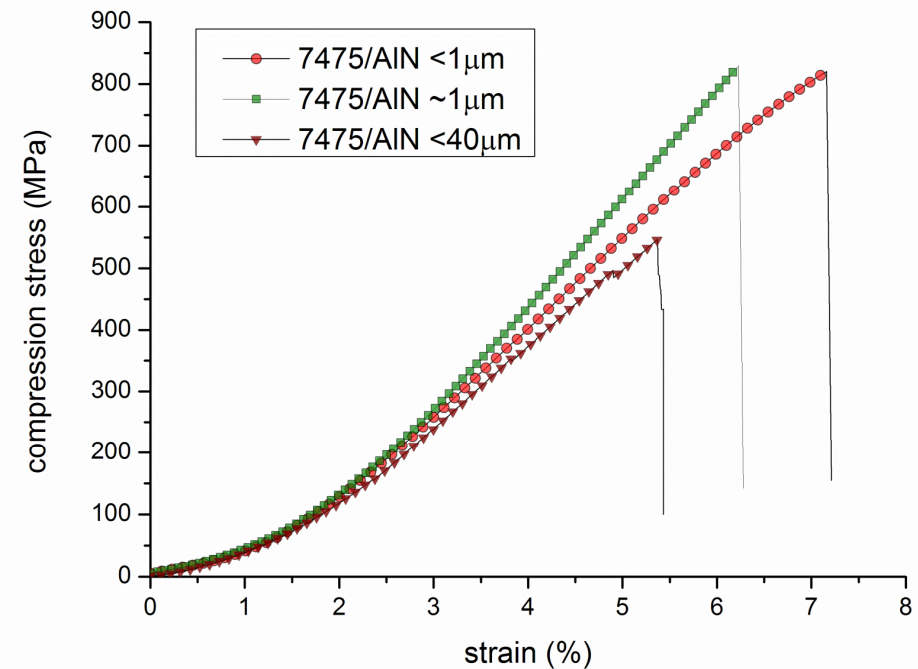
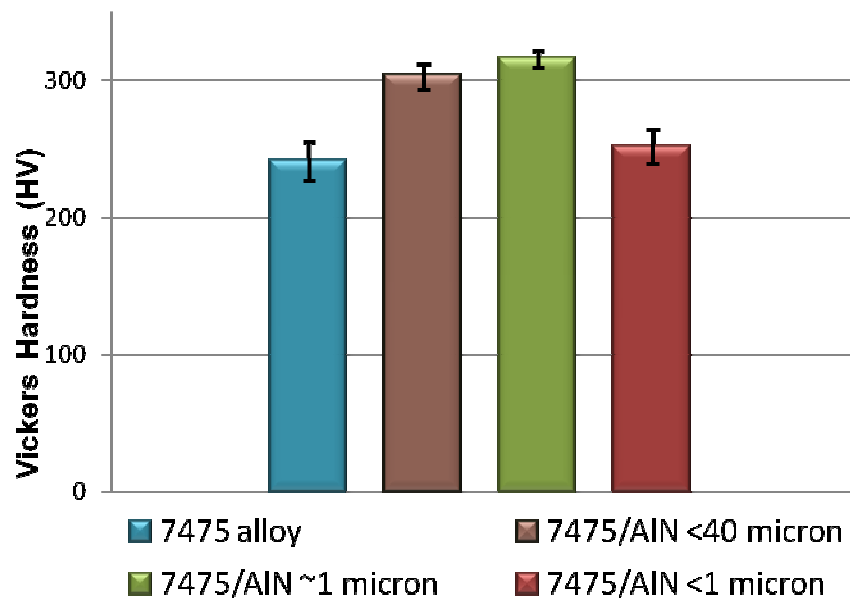


“TEM investigation of metal/ceramic interfaces in AA7475/AlN or Al₂O₃ nano-composites” M. Gajewska, J. Dutkiewicz, L. Lityńska-Dobrzyńska, J. Morgiel, Solid State Phenomena Vol. 186 (2012) pp 202-205



Composite characterization (3):

Mechanical properties



“Microstructure and mechanical properties of AA7475/AlN compacts with varied reinforcing particles size”, M. Gajewska, J. Dutkiewicz, J. Morgiel, accepted for publication in COMPOSITES THEORY AND PRACTICE

„Effect of reinforcement particle size on microstructure and mechanical behavior of AlZnMgCu/AlN nano-composites produced using mechanical alloying” submitted to Journal of Alloys and Compounds



Summary/Preliminary conclusions

- ball milling of 7475 aluminium alloy powder with AlN reinforcement for 40 h allowed to reduce matrix crystallite size down to about ~20 nm
- hot pressing of ball-milled 7475 aluminium alloy powder with AlN particles at 380°C/600MPa allowed to obtain good quality composite samples with retained nanocrystalline matrix
- composites with <math><40\ \mu\text{m}</math> and ~1 μm AlN addition show a **good dispersion of ceramic phase**
- EDS elemental mapping indicated presence of an Mg and O-rich phase at AlN/7475 interfaces
- the best mechanical properties were obtained using 1 micron AlN powder - these composites showed up to **40% hardness improvement and 30% compressive strength improvement** over that of the matrix