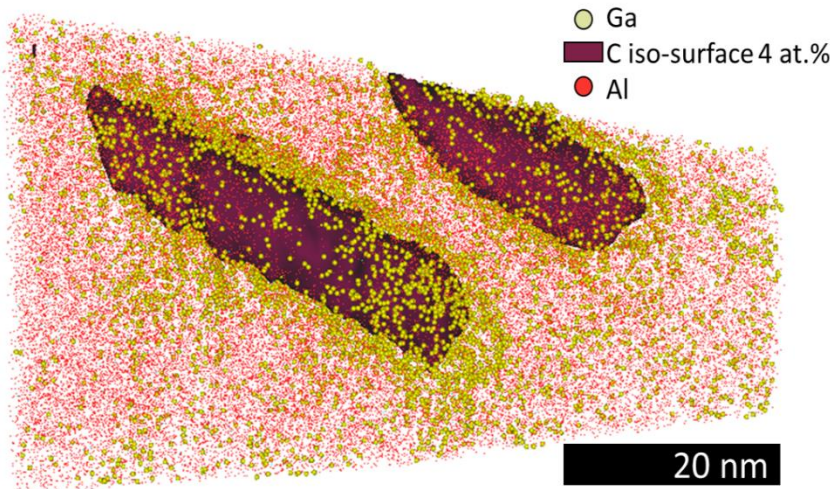


# Diamantane particles in cryomilled nanocrystalline aluminium

Torben Boll, Martin Heilmaier, James Earthman

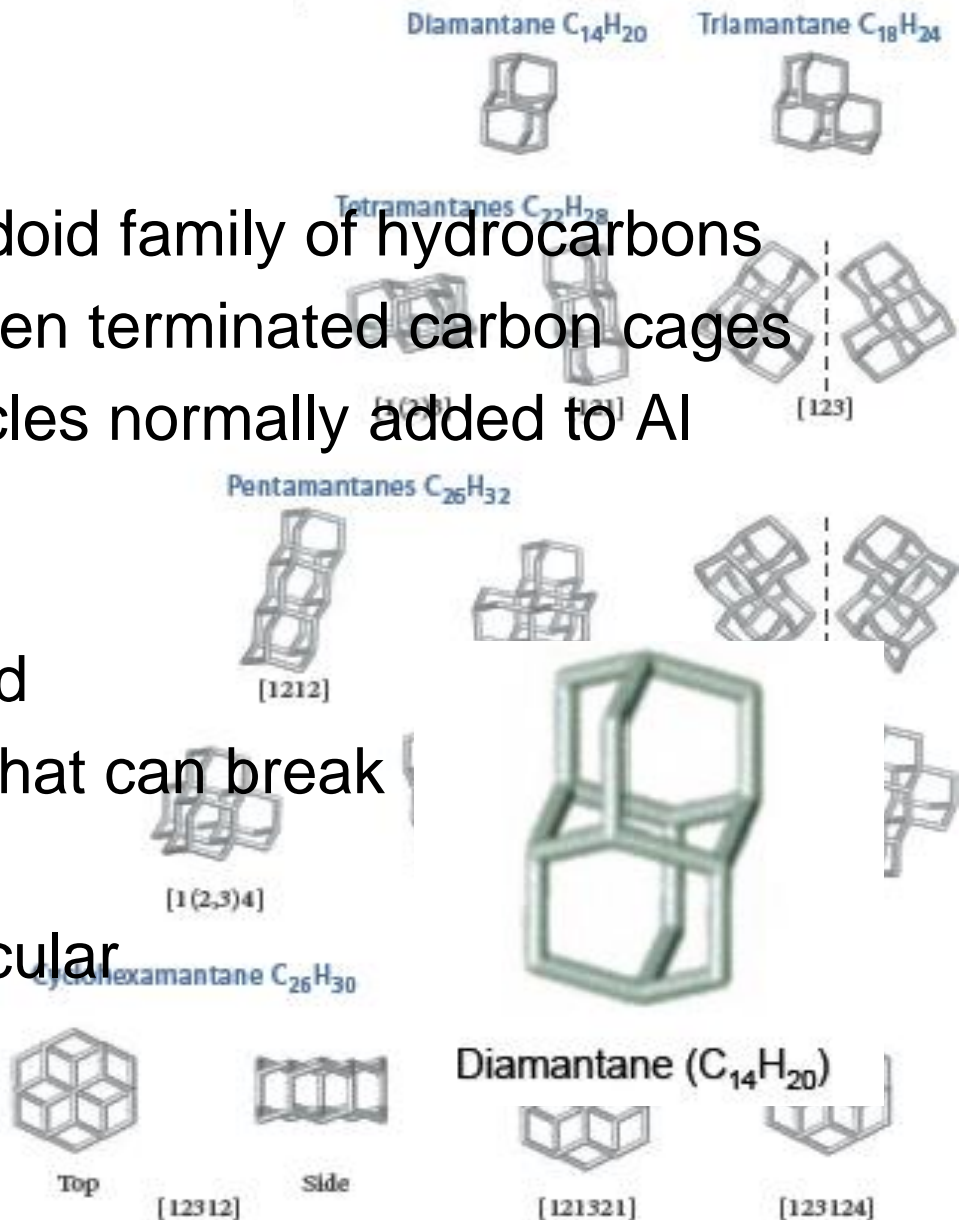


- Background diamantane
- Sample prep: Cryo milling, thermal exposure
- XRD, TEM: Grain growth
- APT: Diamantane (com-)position
- Influence of laser & voltage
- Conclusions

03.10.2017

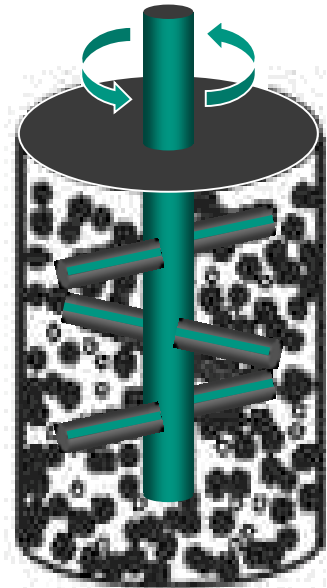
# Diamantane Molecules

- Member of the diamondoid family of hydrocarbons
- Diamond-cubic, hydrogen terminated carbon cages
- Differs from other particles normally added to Al
  - Much smaller (<1 nm vs. ~100 nm)
  - Molecules weakly held together by H bonds that can break up during cryomilling
- Source: Chevron Molecular Diamond Technologies



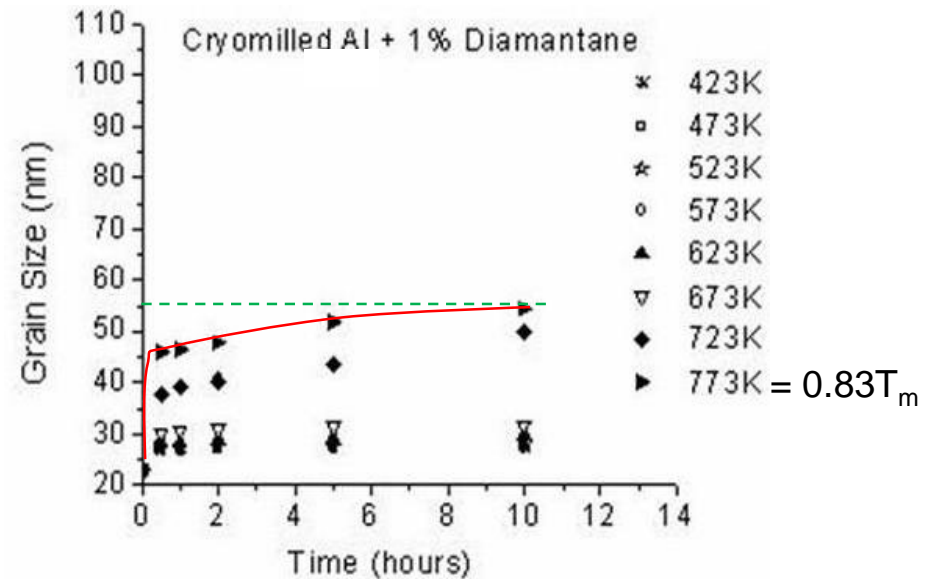
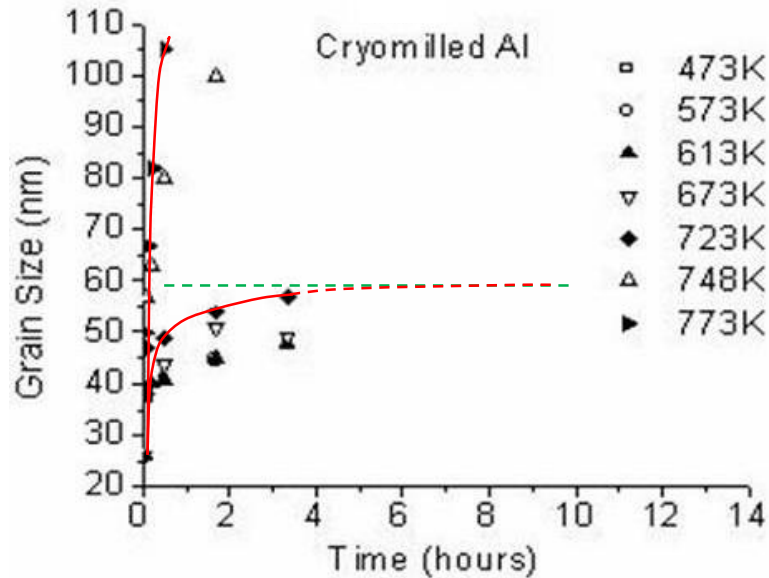
# Cryomilling

- Thermal stability limited -> cryo milling
- Powders milled with steel balls in liquid nitrogen
  - 8 hours at 180 rpm
  - 31:1 (ball:powder) weight ratio
- Diamantane added prior to milling
  - 1wt% is sufficient to cover the grain boundaries for a grain size of 20 nm
- Outgassing in Al canisters
- Hot Isostatic Press (HIP)
  - Pressure of 103 MPa at 693K or 793K
  - 2 hr



# Thermal stability

- Commercially Pure Al (Zhou et al. 2001)
  - Above 748K, mean grain size:  $d > 100$  nm
  - Contains oxides, nitrides, carbides
- CP Al + diamantane (Maung et al. 2011)
  - Substantially reduced grain growth



# Thermal stability

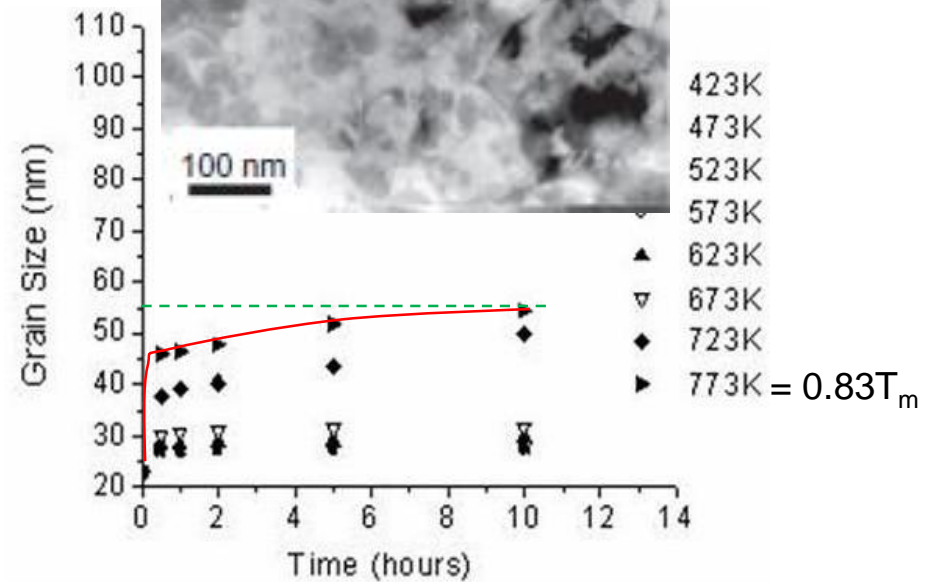
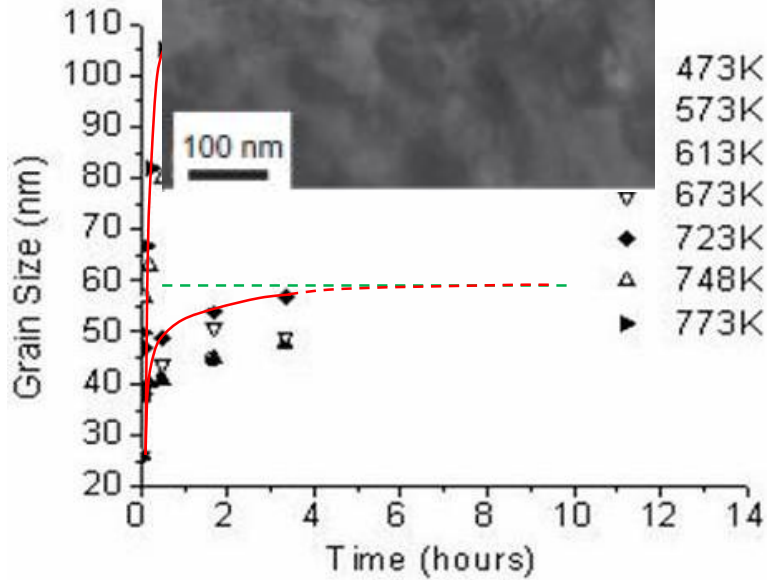
HIP: 103 MPa, 2h, 793 K

$d = 155 \text{ nm}$

$d = 75 \text{ nm}$

Without  
Diamantane

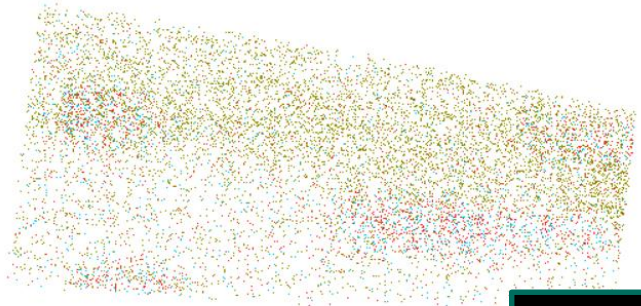
0.5%  
Diamantane



Maung et al. *Acta Materialia* (2012).

# APT: Overview of 4 material types (after HIP)

Al without addition



20 nm

Al + 1wt.% diamantane

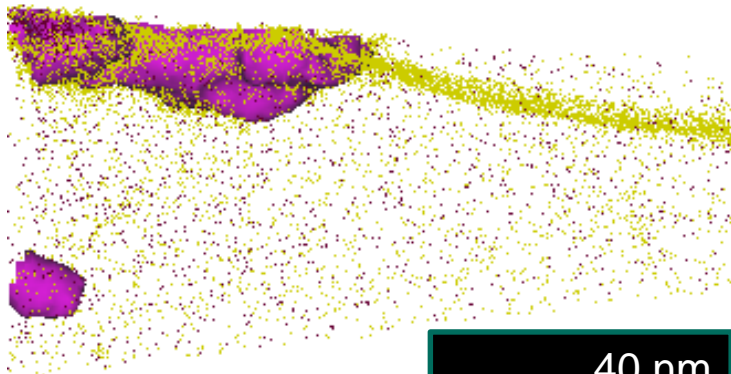


30 nm

- Ga
- C
- Al

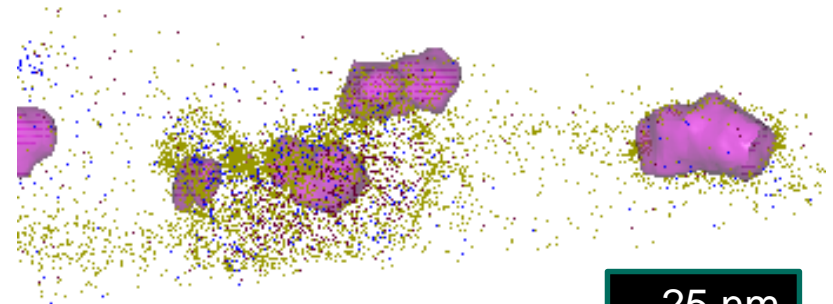
13at.%Mg

Al + 4 wt.%Mg



40 nm

Al +4 wt.%Mg + 1wt.% diamantane



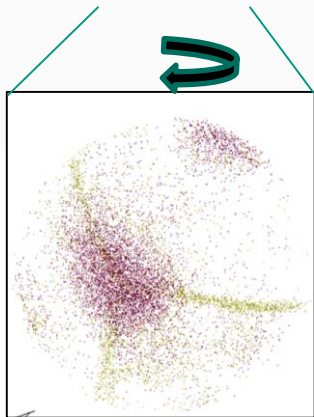
25 nm

# A more detailed look at the positions

Al + 1wt.% diamantane

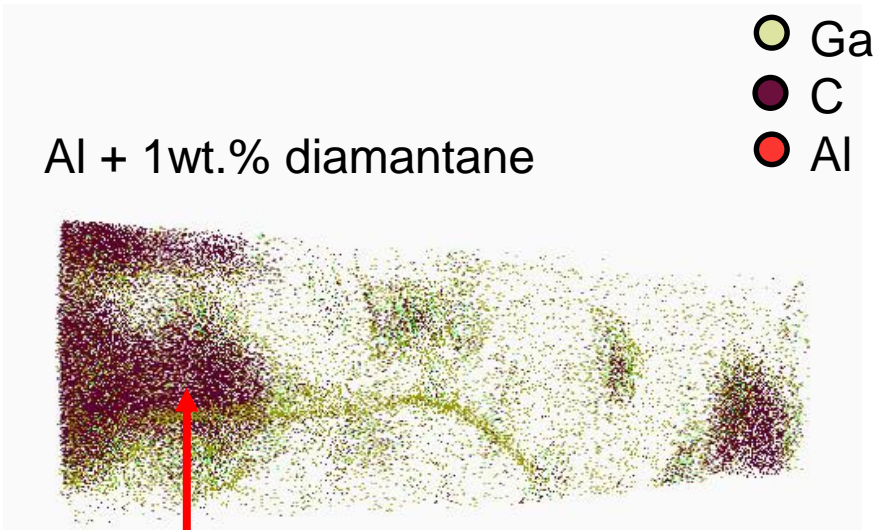
- Ga
- C
- Al

- AlN at GBs
- Diamatane at triple junction



30 nm

# A more detailed look at the compositions



- AlN at GBs
- Diamantane at triple junction
- Oxide/Nitride around diamantane
- No excessive local magnification effect

Proxiaram - Interface 4

T=30K	Det. Rate	Al	C	H	N	O
Laser: 50 pJ	0.2%	45	18	30	0.4	0.4
	1%	60	16	22	0.5	1
	2%	58	26	14	1	1.2
Voltage fraction 15 %	0.2%	34	20	38	4	2



# Conclusions

- Diamantane in cryomilled NC Al and Mg alloys: Remarkable thermal stability.
- Consolidation of bulk NC alloys with  $D < 100$  nm.
- Mg had less influence on grain growth mechanism in the presence of diamantane.
  
- Diamantane runs surprisingly well in APT
- Diamantane at grain boundaries and especially triple junctions
- Mg, AlN, AlO in separate clusters along the GBs
- Diamantane clusters contain Al,N,O

# Thank you for your attention

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KNMF grants APT time to suitable projects