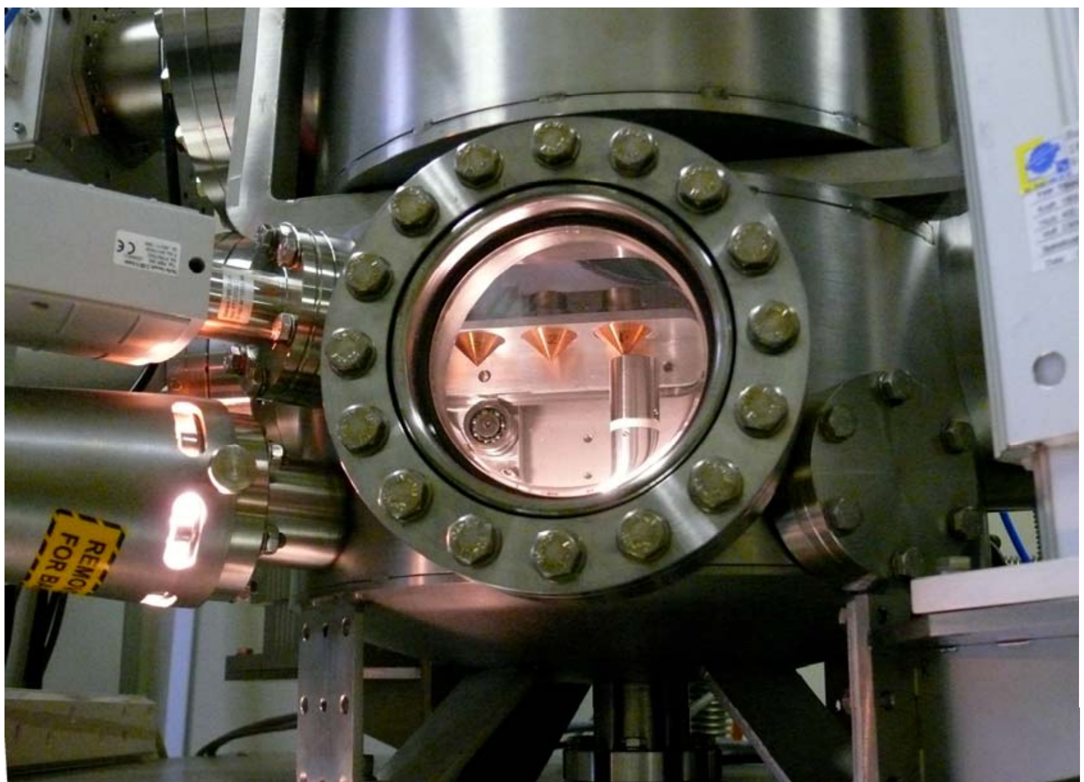
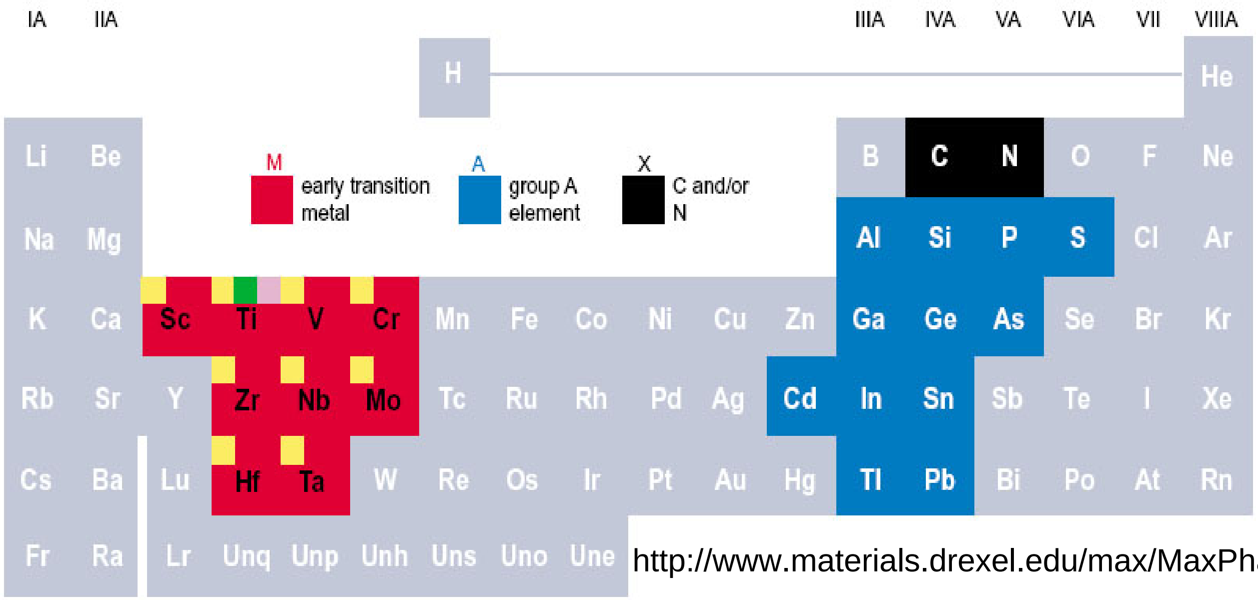
Synthesis and compositional analysis of thin film $M_{n+1}AX_n$ phases

M. Guenette, M. Tucker, Y.Yin, M.M.M.Bilek, D.R.McKenzie

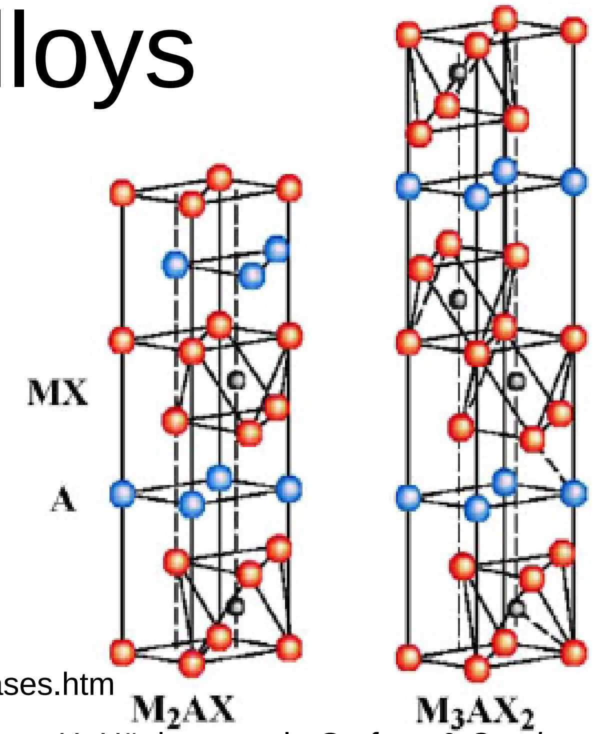
School Of Physics, University Of Sydney



$M_{n+1}AX_n$ Phase Alloys



http://www.materials.drexel.edu/max/MaxPhases.htm



H. Högberg et al. *Surface & Coatings Technology* 193: 6–10, 2005

MAX phases exhibit a unique combination of ceramic and metallic properties

<u>Ceramic</u>	<u>Metallic</u>
• Stiff	• Machinable
• Oxidation resistant	• Resistant to thermal shock
• High temperature stability	• High thermal and electrical conductivity

Barsoum, M. W. *Progress in Solid State Chemistry,* 28(1-4): 201:281, 2000

Methods of Synthesis

Hot sintering[1]

- Powders pressed into bulk form at high temperature (>1500^{o}C) and pressure (~50MpA)
- Contains high levels of impurities and low phase purity, polycrystaline

DC magnetron sputtering[2]

- Compound or multiple targets to synthesise single crystal thin films
- Substrate temperature ~900^{o}C

Pulsed Cathodic Arc[3]

- Multiple cathode to synthesise single crystal thin films
- Substrate temperature ~900^{o}C

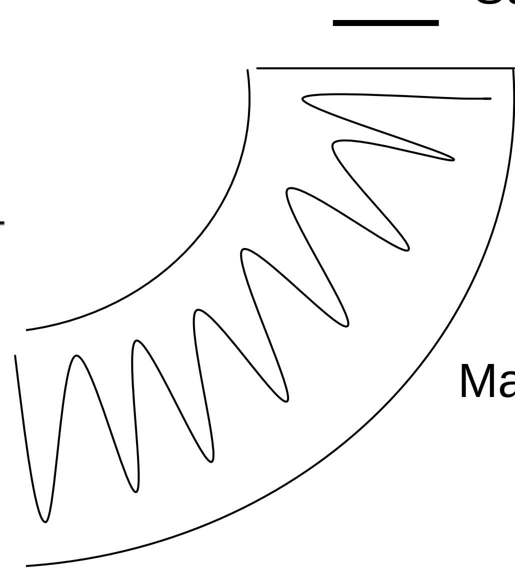
[1] Barsoum, M. et al, Journal of the American Ceramic Society , 82: 2545 1999
[2] Palmquist, JP. et al, Applied Physics Letters, 81 (5): 835-837 2002
[3] Rosen. J. et al, Journal of Applied Physics, 101 (5): 2007

Pulsed Cathodic Arc

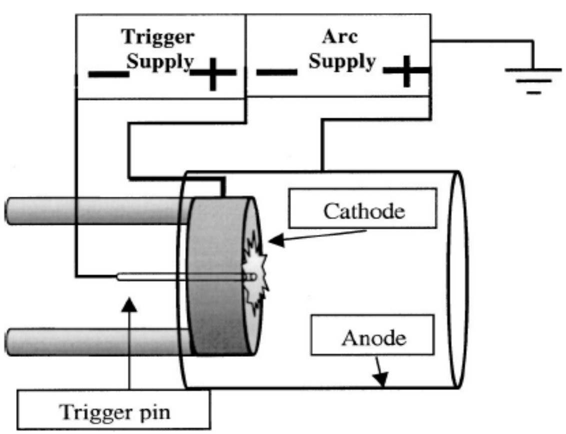
- Stable, reproducible plasma density from 3 cathodes, M, A and X
- Can control number of plasma pulses from each elemental cathode and control amount of each element reaching the substrate
- Linear relationship between the number of pulses, P_x and amount of element reaching the substrate, N_x is expected.

$$\frac{N_x}{N_y} = \frac{A_x}{A_y}\frac{P_x}{P_y}$$

Substrate and Heater



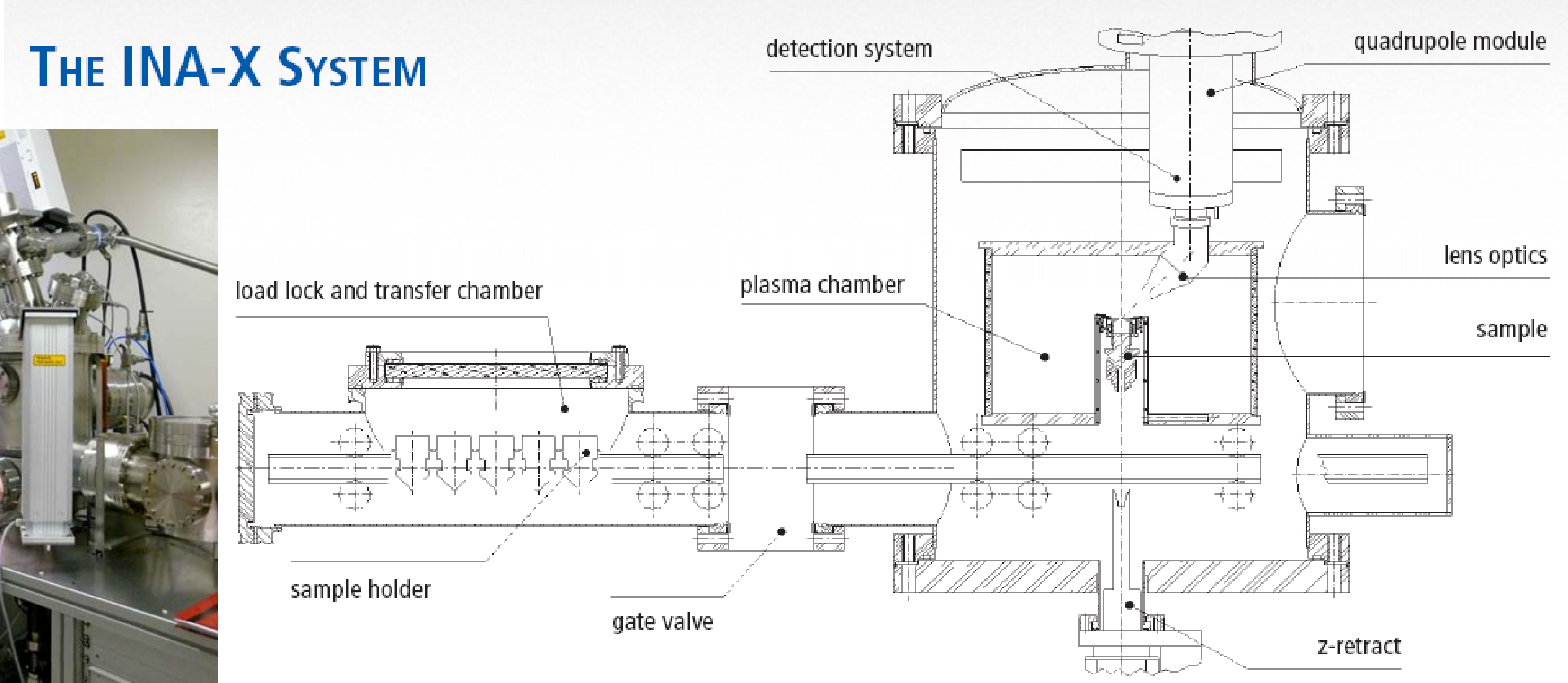
Magnetic macroparticle filter



Cathode/Anode assembly x 3

Oates, T. W. H. et al, Review Of Scientific Instruments, 74(11), 4750, 2003

Secondary Neutral Mass Spectrometry

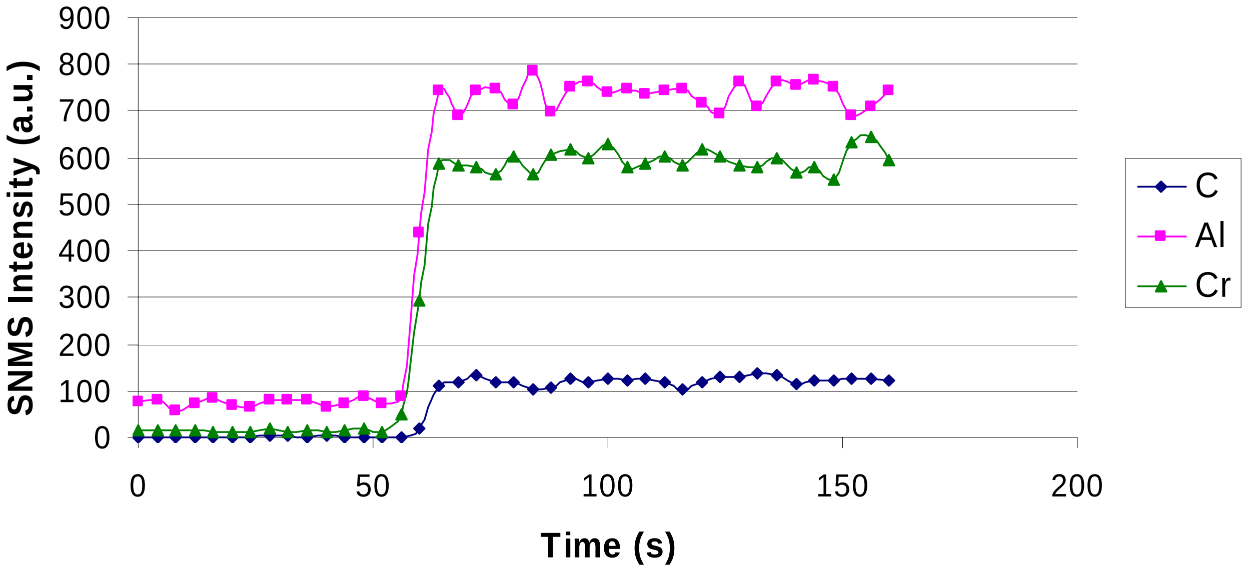


- Sample sputtered with Ar Plasma
- Emerging neutral particles are post-ionised and measured with mass spectrometer

SNMS Analysis

The elemental concentration ratio, C_x/C_y in the sputtered sample is directly proportional to the ratio of the SNMS signal intensity, I_x/I_y

SNMS Time profile for Cr-Al-C sample



$$\frac{C_x}{C_y} = \frac{I_x}{I_y}\frac{D_x}{D_y}$$

D_x/D_y are relative sensitivity factors that consider geometry, detection and ionisation probability.

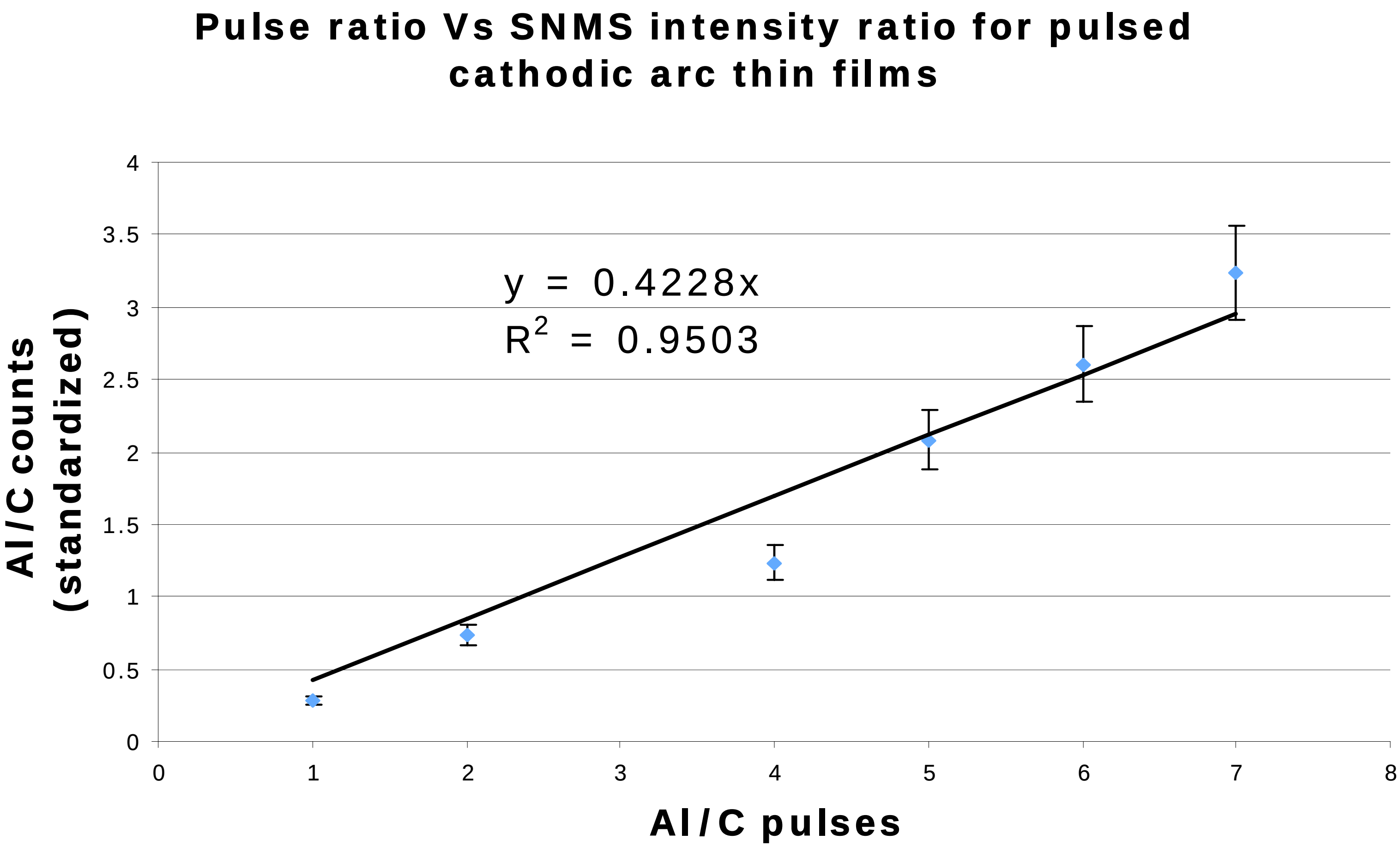
Quantitative composition analysis is possible with standards of known composition

Oechsner, H. et al, Journal of Applied Physics, 14(1), 43-47, 1977

SNMS and Pulsed Cathodic Arc

Combining the theory of SNMS and Pulsed cathodic arc, a linear relationship between pulse ratio and SNMS intensity ratio is expected.

$$\frac{P_x}{P_y} = M_{xy} \frac{I_x}{I_y}$$

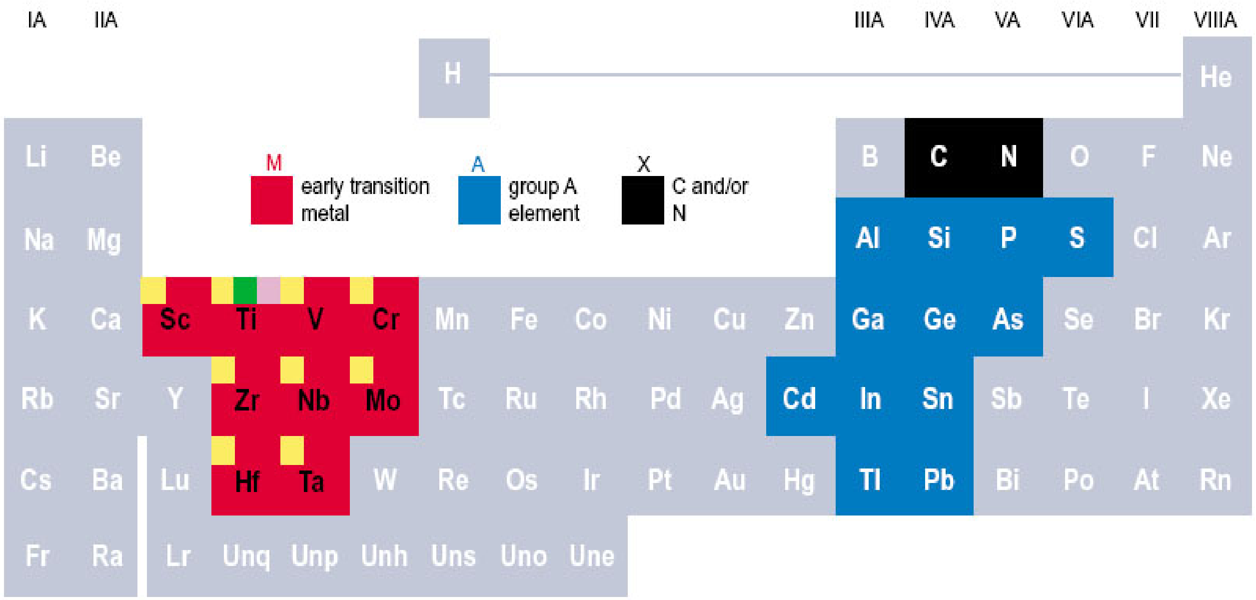


Summary

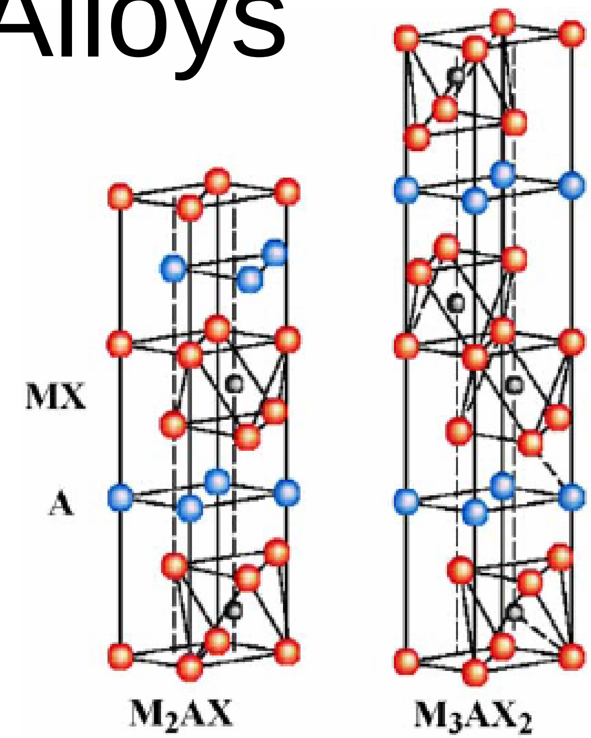
- Using the pulsed cathodic arc combined with SNMS composition analysis, a relationship between pulse ratio and stoichiometric ratio has been established given a set of constant deposition parameters.

- SNMS is a fast and reliable method of accurately controlling and quantifying the composition of thin film MAX phases

Mn+1AXn Phase Alloys



http://www.materials.drexel.edu/max/MaxPhases.htm



H. Högberg et al. *Surface & Coatings Technology* 193: 6–10, 2005

Ceramic

- Stiff, oxidation resistant, retains properties at high temperatures

Metallic

- Machinable, resistant to thermal shock, high thermal and electrical conductivity

Barsoum, M. W. *Progress in Solid State Chemistry,* 28(1-4): 201:281, 2000