Pack Cementation: The Effect of Atmosphere and Substrate On Coating Composition

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Project Overview

- **Pack cementation has been used for 20-30 years to produce protective surface coatings on superalloys in the gas turbine engine industry**

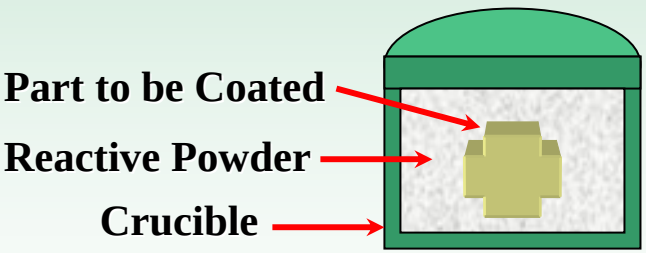
- **Pack cementation is a simple process that produces a uniform coating on all surfaces of a substrate**

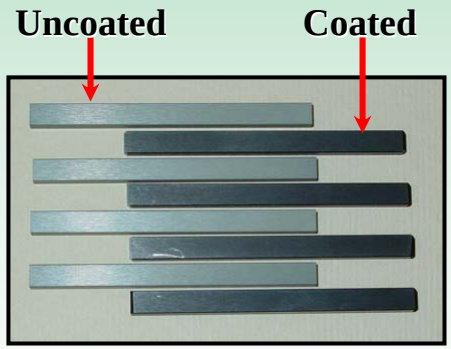
- **Tests have shown that a pack cementation process can be used to form surface coatings on Si_3N_4**

Project Overview

- **Pack cementation coatings may be able to act as stand-alone protection**

- **Pack cementation coatings may be used as a surface preparation or bond coat for subsequent overlay coating**





Pack cementation of AS800 Si_3N_4 bars in an Al - NH_4Cl - Al_2O_3 pack at 1000°C for 5 hr. in Argon

Pack Cementation Processing Variables

- **Substrate Composition – AS800, NT154, SN281**
- **Reactive Powder Bed – $AlCl_3$, $Sr(NO_3)_2$, $Y(NO_3)_3$, $ZrCl_4$**
- **Furnace Atmosphere – Air, Argon, Nitrogen**
- **Processing Temperature – 1200ºC**

Silicon Nitrides for Pack Cementation Coating

Si_3N_4	Additives	Grain Boundary Phase
Honeywell AS800	La, Y, Sr	La-Apatite
Saint Gobain NT154	Y	$Y_2Si_2O_7$
Kyocera SN281	Lu	$Lu_2Si_2O_7$

Coating Phases Identified by X-ray Diffraction Analysis

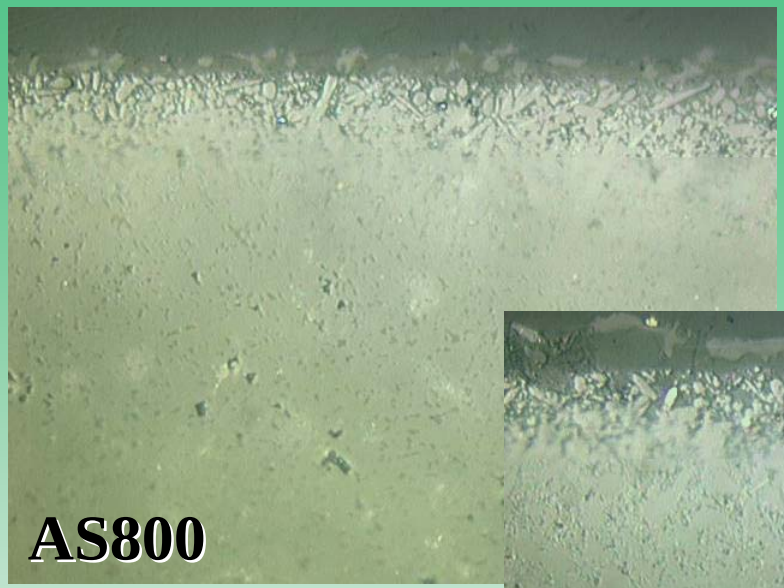
	AS800 (La-Apatite)	NT154 ($Y_2Si_2O_7$)	SN281 ($Lu_2Si_2O_7$)
	Air		
$AlCl_3$	Al_2O_3, ?	Y_2SiO_5	SiO_2, Al_2O_3, ?
$Sr(NO_3)_2$	?	Y_2SiO_5,?	SiO_2, ?, ?
$Y(NO_3)_3$	?	Y_2SiO_5	?, ?
$ZrCl_4$	Al_2O_3, ZrO_2, SiO_2	Al_2O_3, Y_2SiO_5	ZrO_2, Al_2O_3, SiO_2
	Argon		
$AlCl_3$	AlN, Si	Si, YAG?, ?	Si, Lu-Apatite?, AlN?
$Sr(NO_3)_2$	$SrSiO_3$, ?	$SrSiO_3$, $Sr_3Al_2O_6$, ?	?, ?
$Y(NO_3)_3$	$Y_2Si_2O_7$, $Y_4Si_3O_{12}$, ?	$Y_4Si_3O_{12}$, ?, ?	Y_2SiO_5?
$ZrCl_4$	ZrN	ZrN	ZrN, Lu-Apatite?
	Nitrogen		
$AlCl_3$	La-Apatite (weak)	none	Lu_2SiO_5
$Sr(NO_3)_2$	$SrSiO_3$, Sr_2SiO_4, SiO_2	$SrSiO_3$, $Sr_3Al_2O_6$, ?	$SrSiO_3$, ?
$Y(NO_3)_3$	$Y_2Si_2O_7$	?	$Y_2Si_2O_7$, Lu_2SiO_5
$ZrCl_4$	$ZrSiO_4$, SiO_2	$ZrSiO_4$	$ZrSiO_4$

Examples of the Effect of Varying Silicon Nitride Substrate

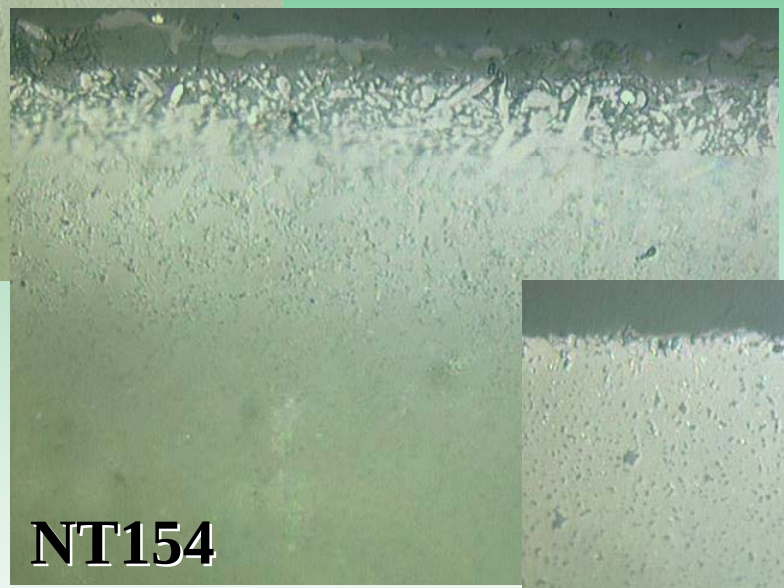
- Optical micrographs of coated sample cross-sections showing coating morphology
- Identification of coating phase compounds

Si_3N_4 Substrate Effects

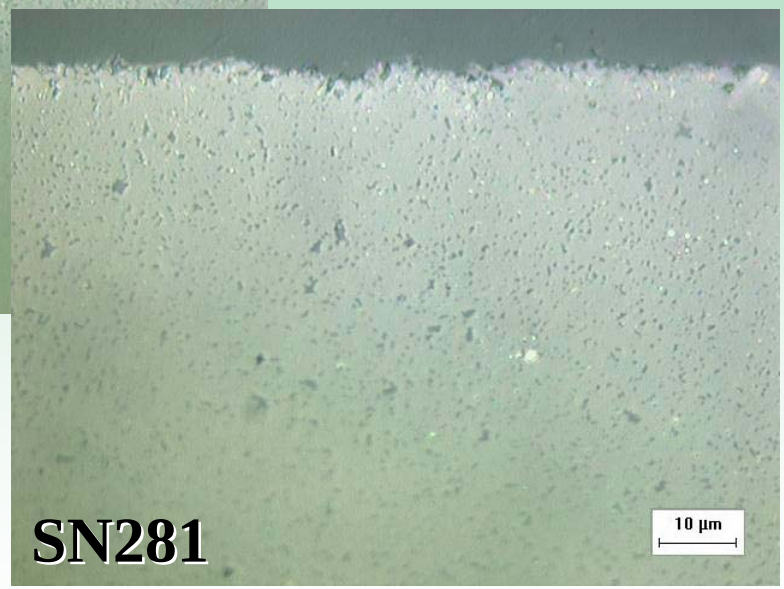
$Sr(NO_3)_2$, 1200°C, Ar



Coating phases: $SrSiO_3$, ?, ?



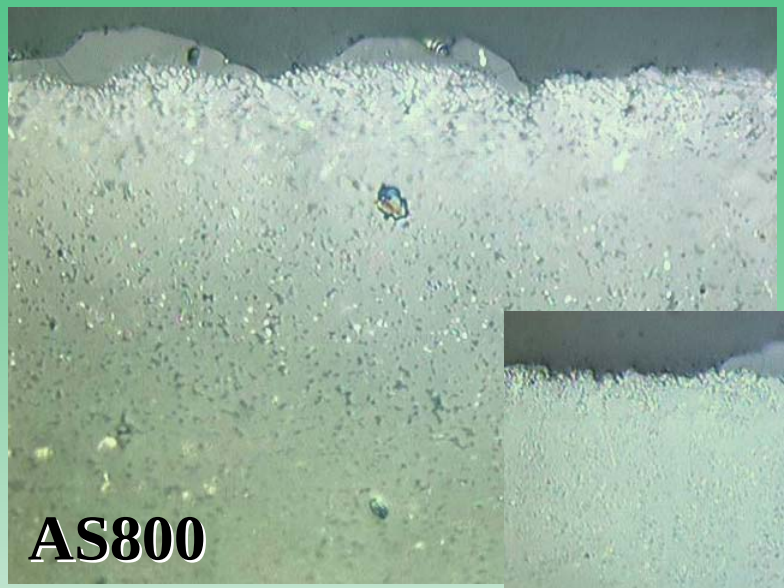
Coating phases: $SrSiO_3$, $Sr_3Al_2O_6$, ?



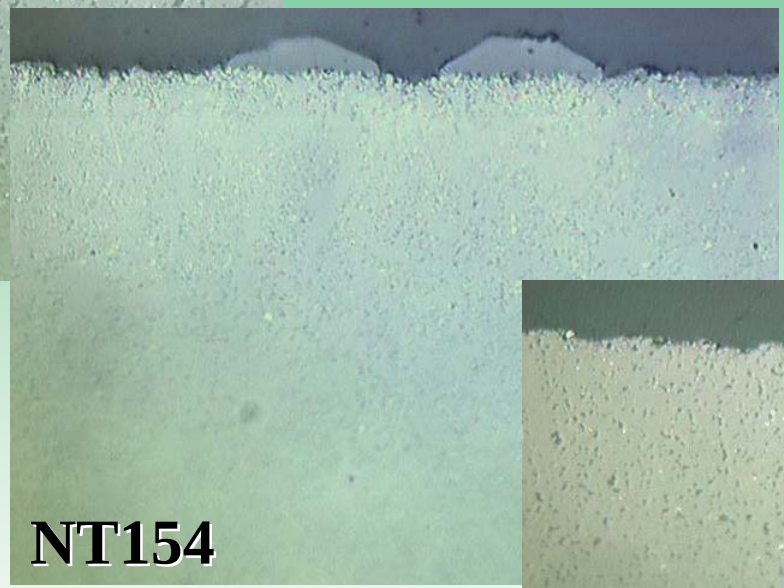
Coating phases: ?, ?

Si_3N_4 Substrate Effects

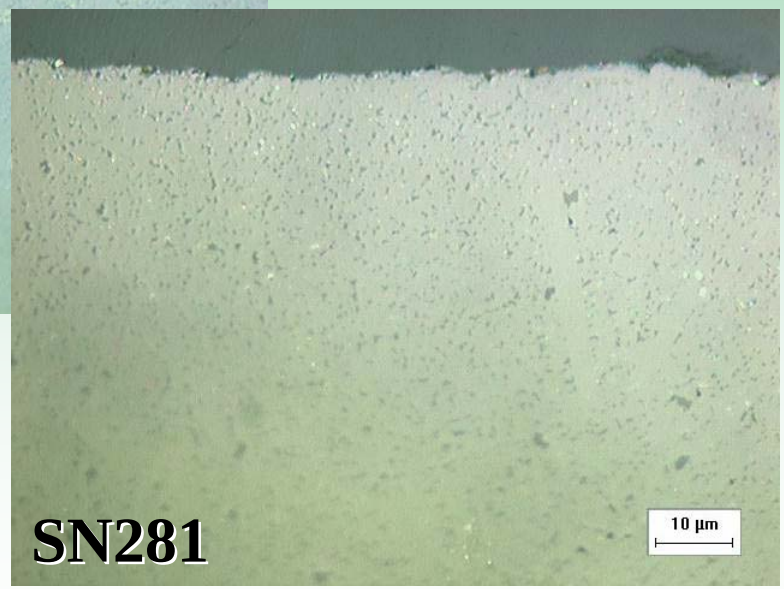
$Y(NO_3)_3$, 1200°C, Ar



Coating phases:
$Y_4Si_3O_{12}$, ?



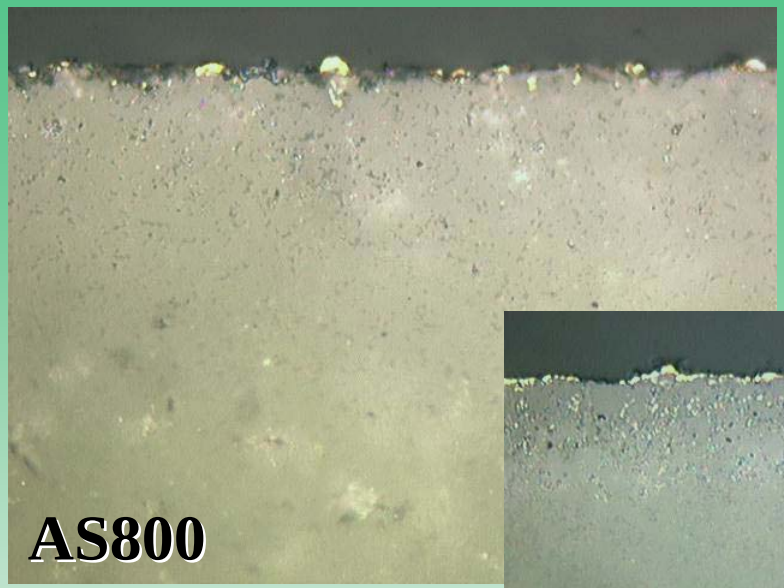
Coating phases: $Y_4Si_3O_{12}$, ?



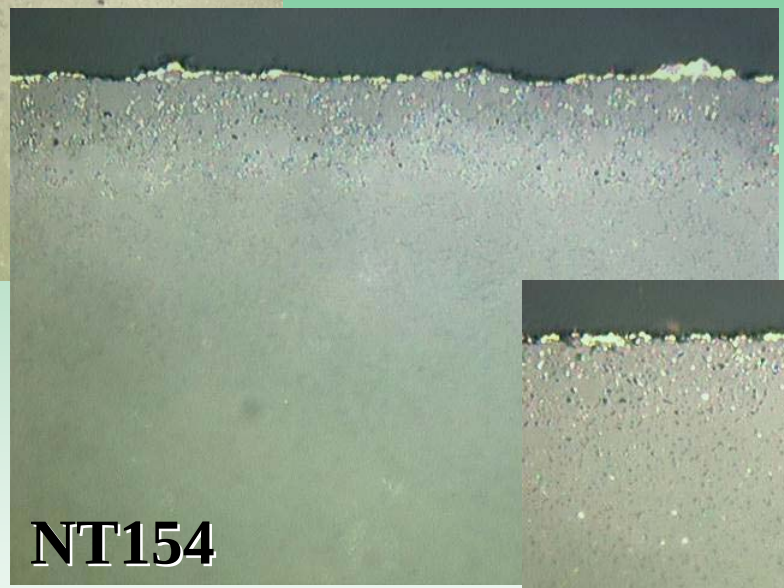
Coating phases: Y_2SiO_5

Si_3N_4 Substrate Effects

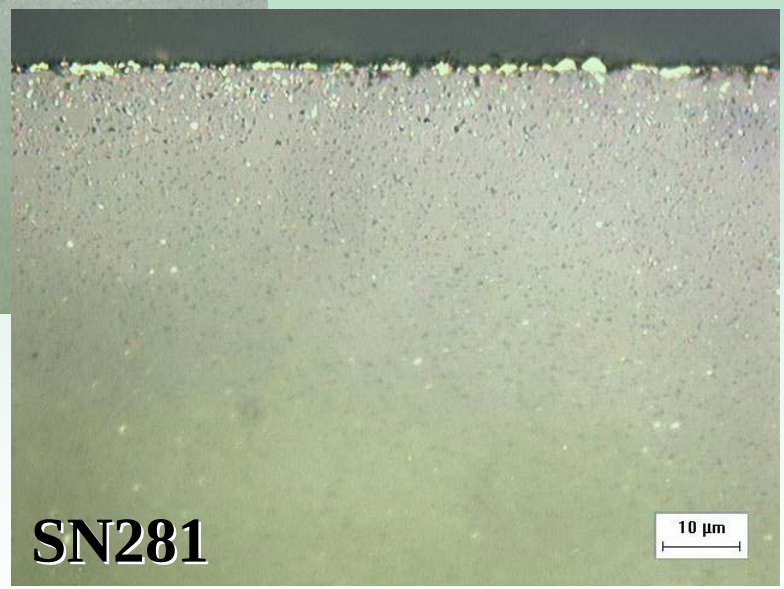
$ZrCl_4$, 1200°C, Ar



Coating phases: ZrN



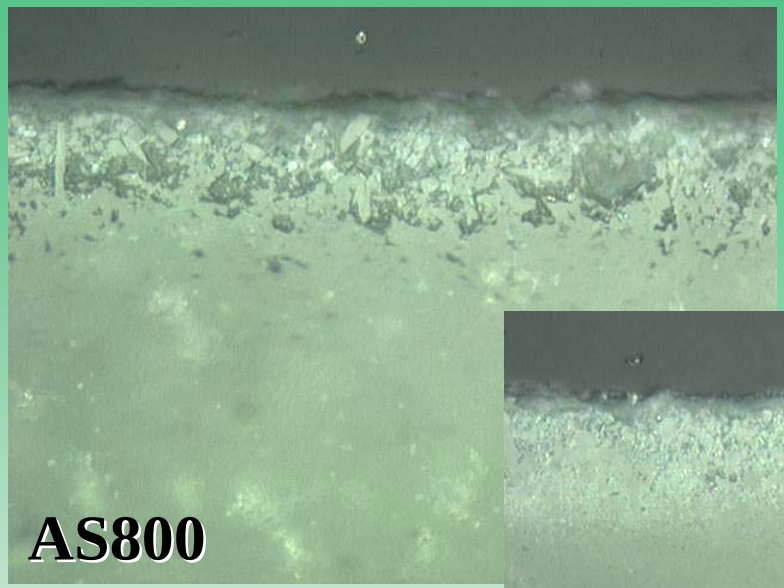
Coating phases: ZrN



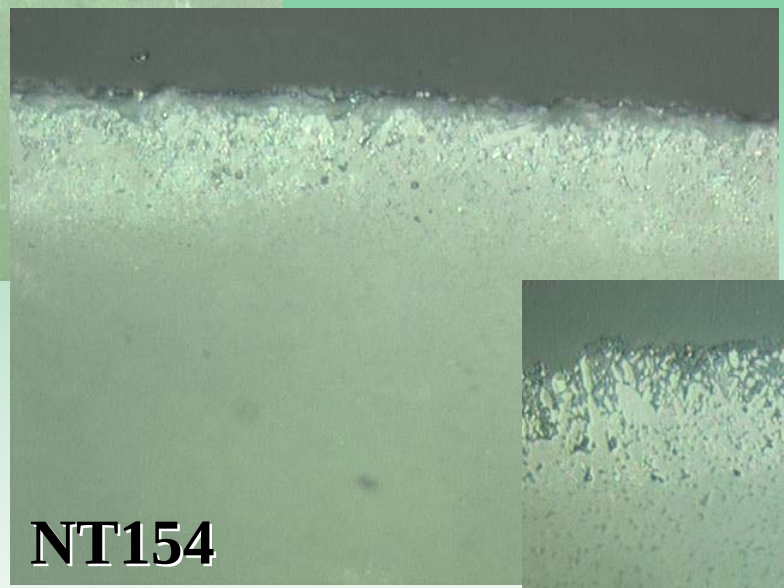
Coating phases: ZrN

Si_3N_4 Substrate Effects

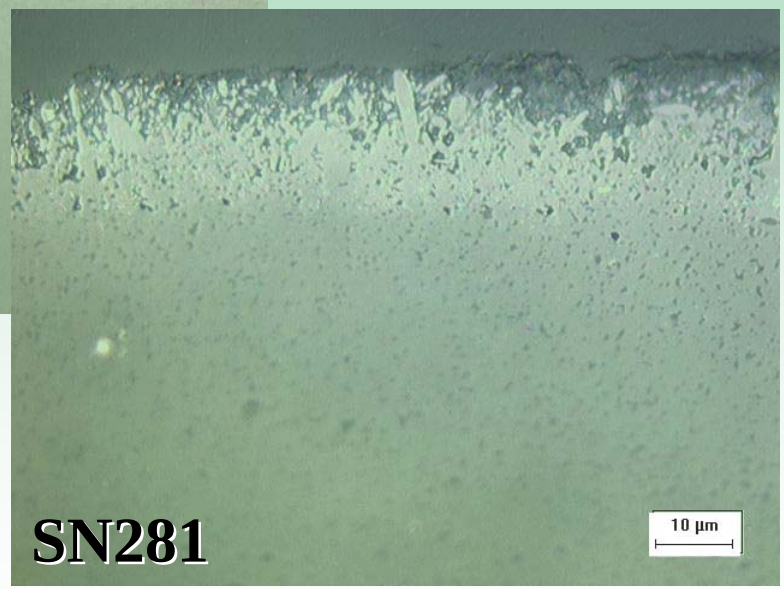
$Sr(NO_3)_2$, 1200°C, N_2



Coating phases: $SrSiO_3$, Sr_2SiO_4, SiO_2



Coating phases: $SrSiO_3$, $Sr_3Al_2O_6$, ?



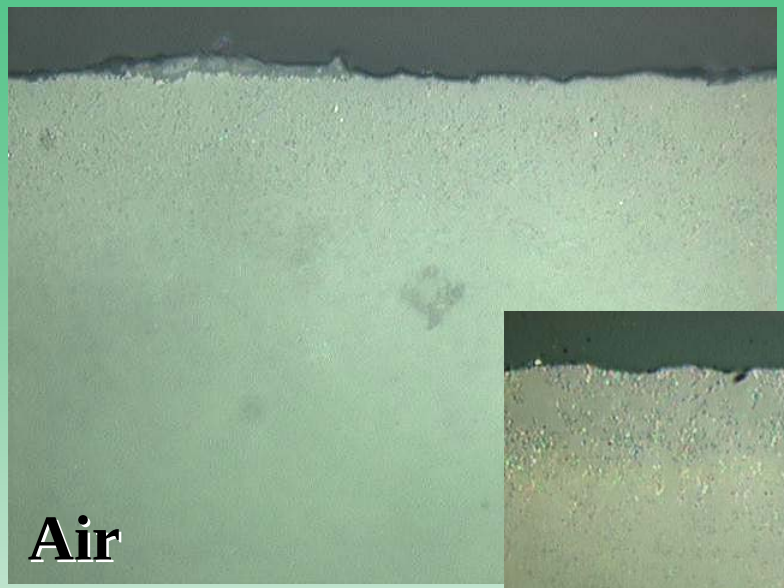
Coating phases: $SrSiO_3$, ?

Examples of the Effect of Varying Furnace Atmosphere

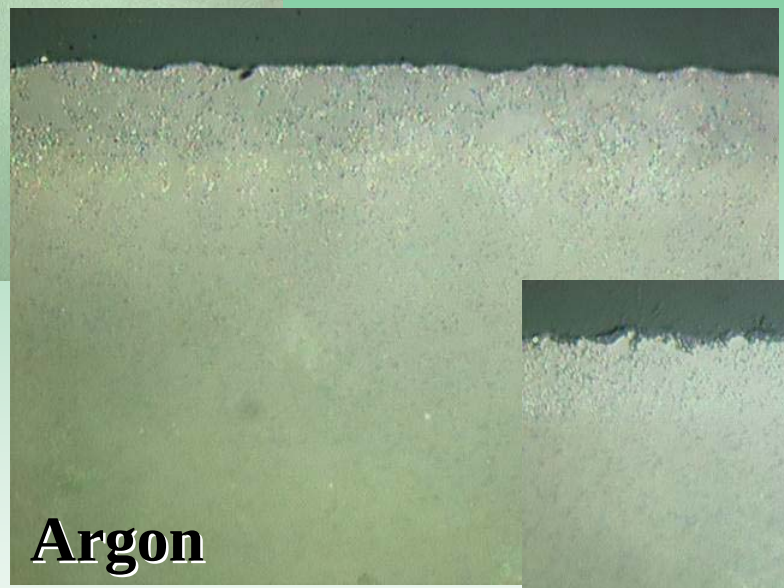
- Optical micrographs of coated sample cross-sections showing coating morphology
- Identification of coating phase compounds

Furnace Atmosphere Effects

NT154, $AlCl_3$, 1200°C



Coating phases:
Y_2SiO_5

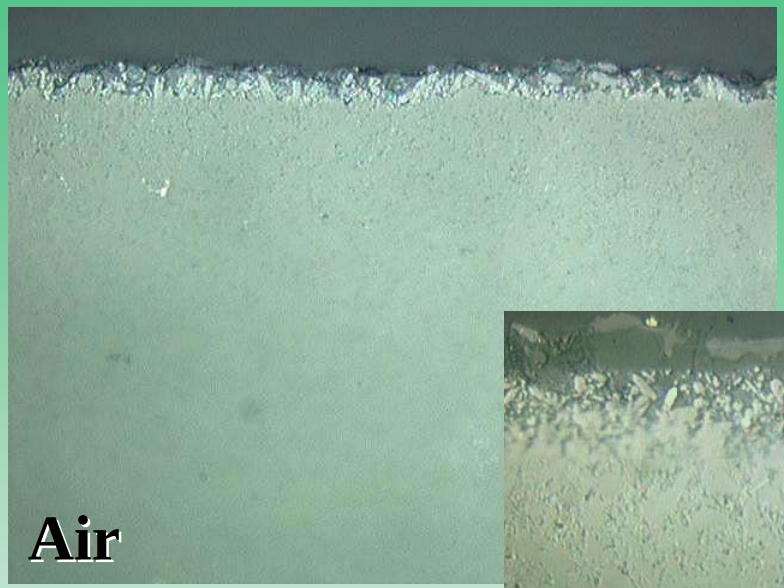


Coating phases: Si, YAG?

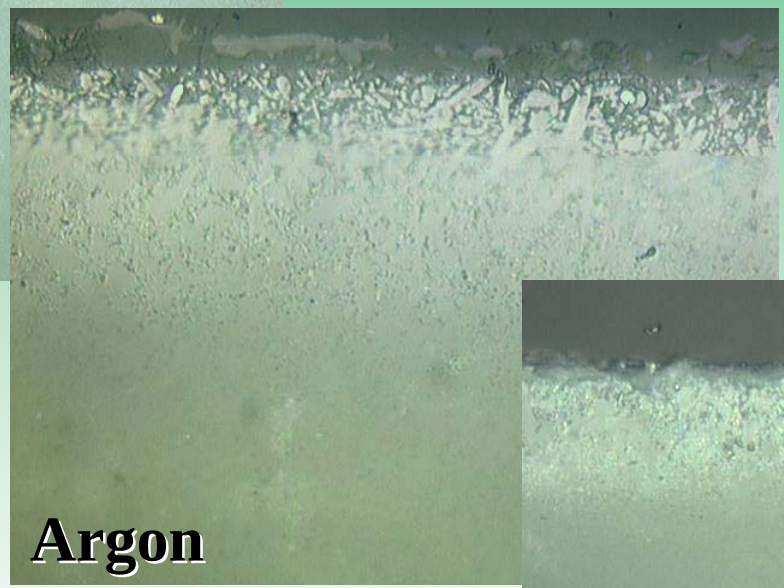
Coating phases: none

Furnace Atmosphere Effects

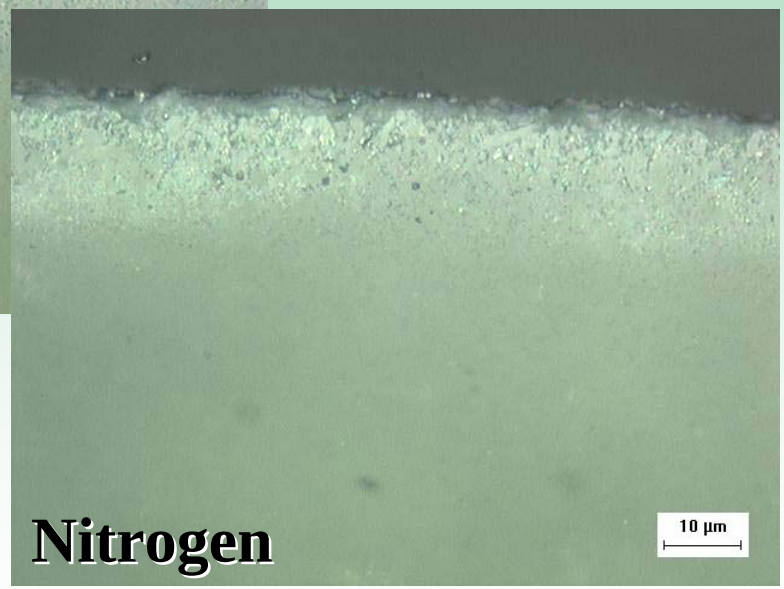
NT154, $Sr(NO_3)_2$, 1200°C



Coating phases:
Y_2SiO_5, ?



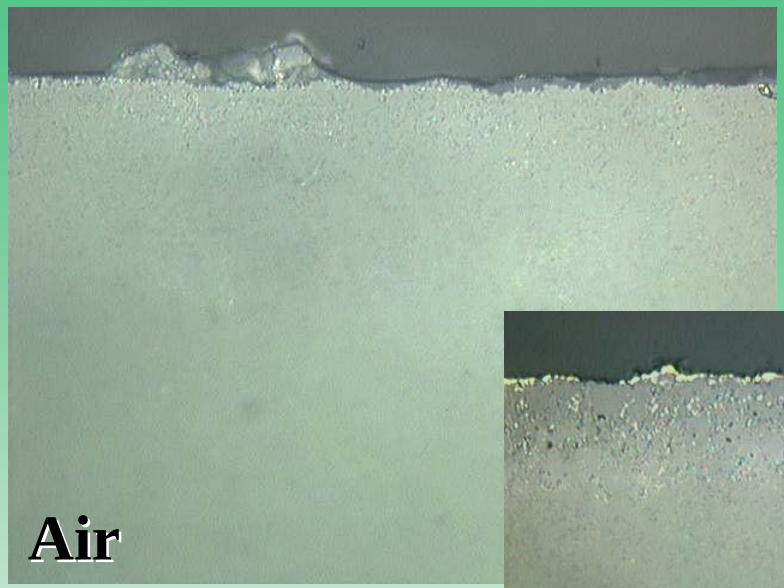
Coating phases: $SrSiO_3$, $Sr_3Al_2O_6$, ?



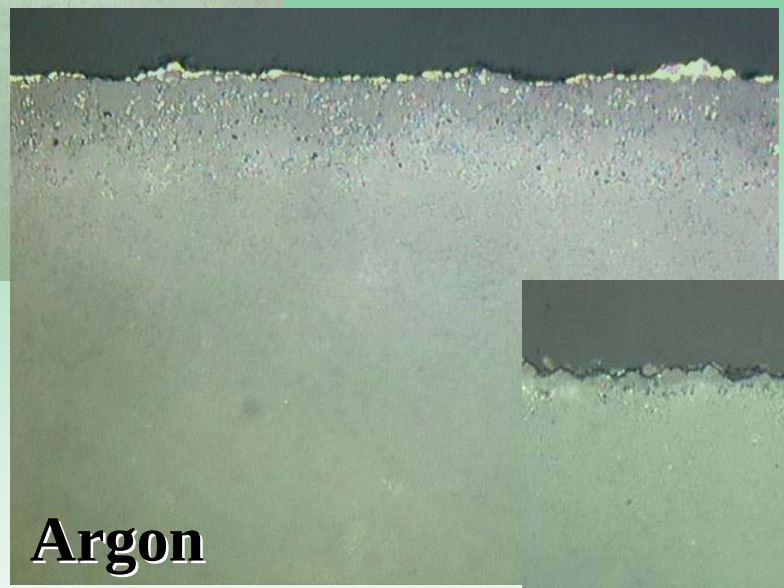
Coating phases:
$SrSiO_3$, $Sr_3Al_2O_6$, ?

Furnace Atmosphere Effects

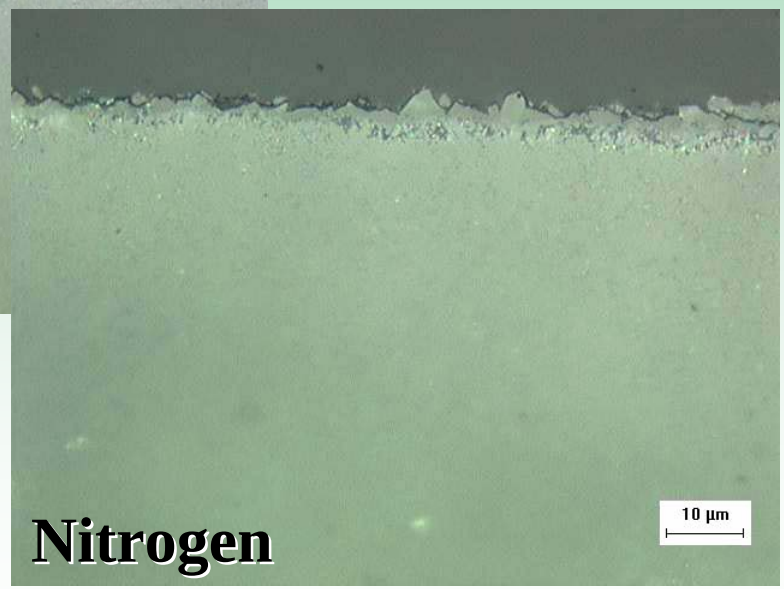
NT154, $ZrCl_4$, 1200°C



Coating phases:
Al_2O_3, Y_2SiO_5



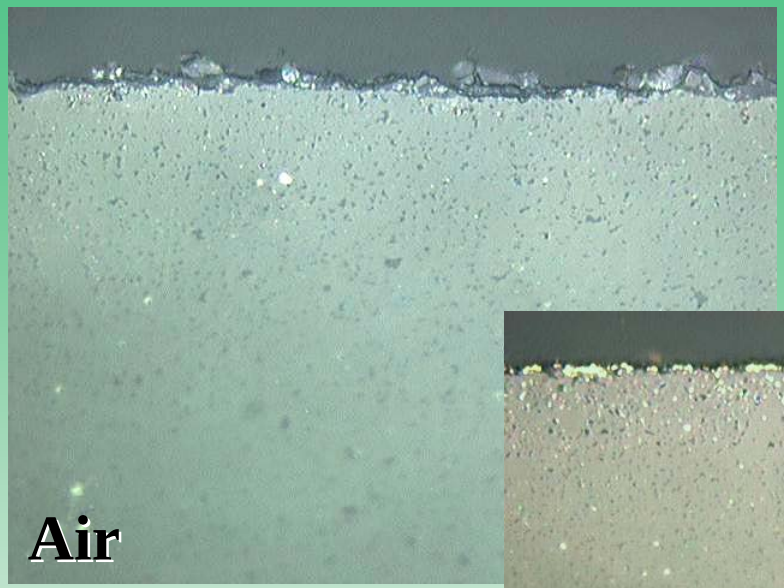
Coating phases: ZrN



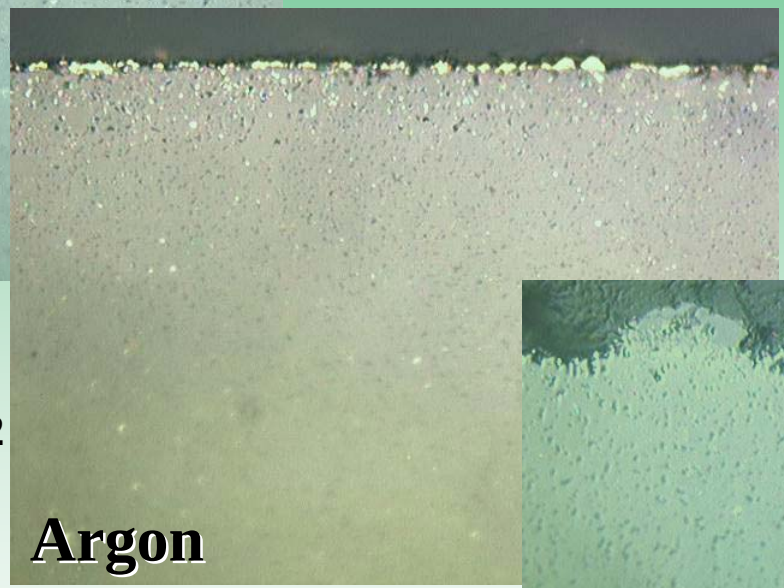
Coating phases: $ZrSiO_4$

Furnace Atmosphere Effects

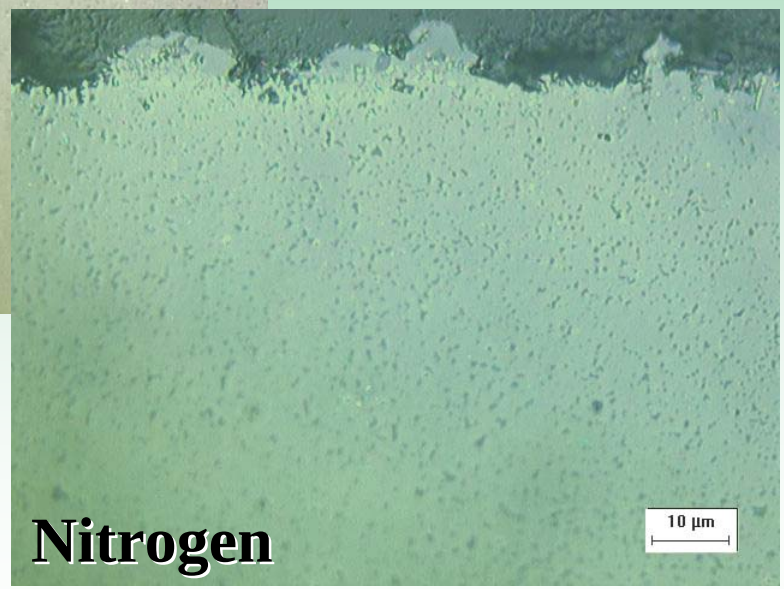
SN281, $ZrCl_4$, 1200°C



Coating phases:
ZrO_2, Al_2O_3, SiO_2



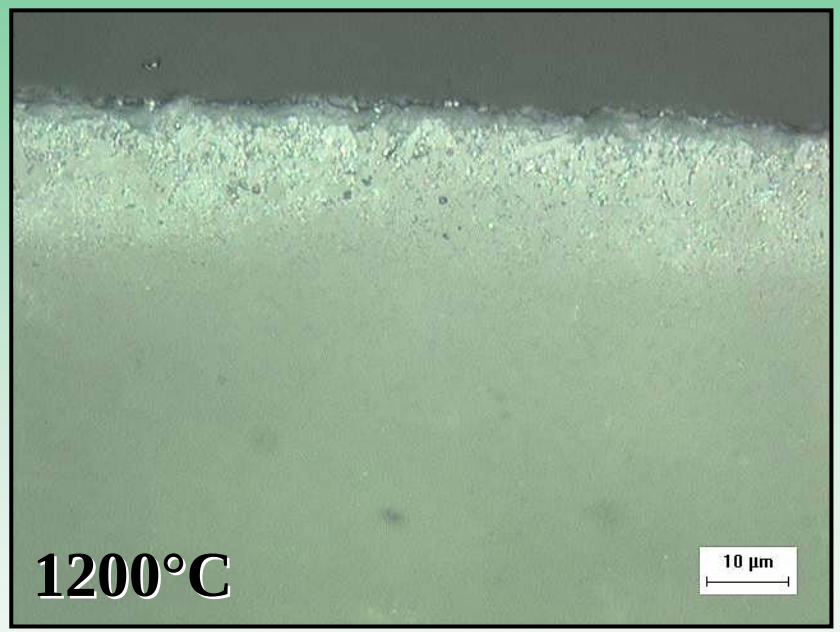
Coating phases: ZrN



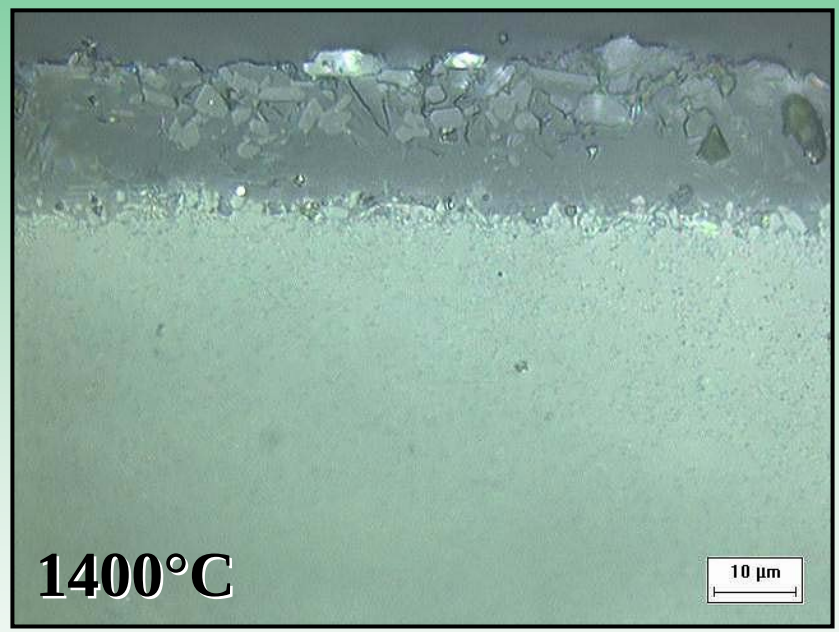
Coating phases: $ZrSiO_4$

Effect of Processing Temperature

NT154, $Sr(NO_3)_2$, N_2



$SrSiO_3$, $Sr_3Al_2O_6$, ?



$SrAl_2Si_2O_8$

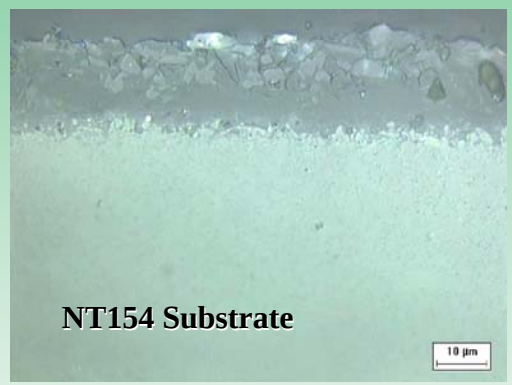
Summary

- Pack cementation coatings were formed on three different Si_3N_4 substrates in three different furnace atmospheres
- Both the substrate and the furnace atmosphere had an effect on the resulting coating for most of the pack compositions that were tested
- Results show promise that pack cementation may be developed to form stand-alone protective coatings or bond coats on Si_3N_4

FY 2004 Plans

Pack Cementation for EBCs:

- Focus on producing promising coating compounds on various Si_3N_4 substrates
  - $SrAl_2O_4$
  - $SrAl_2Si_2O_8$
  - $Yb_2Si_2O_7$



- Conduct exposure tests to evaluate protective properties of coatings in a simulated turbine engine environment