

Low-temperature coatings for instruments on the BepiColombo mission to Mercury

Anna-Lena Larsson, Jan-Erik Wahlund

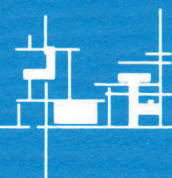
IRF Technical Report 049
July 2004

ISSN 0284-1738

INSTITUTET FÖR RYMDFYSIK

Swedish Institute of Space Physics

Kiruna, Sweden



Low-temperature coatings for instruments on the BepiColombo mission to Mercury

*IRF Technical Report 049
July 2004*

Anna-Lena Larsson, Jan-Erik Wahlund

ISSN 0284-1738

Printed in Sweden
Swedish Institute of Space Physics
Kiruna, Sweden, 2004

Contents

1	Introduction	2
2	Background.....	4
2.1	Heat transfer in a Mercury environment	4
2.2	Optical properties of materials	4
2.2.1	Absorptance and emittance	4
2.2.2	Thin film interference	5
2.3	Environmental conditions.....	6
2.4	Surface coatings requirements.....	6
2.5	Materials selection.....	6
2.5.1	Thin film TiN and TiAlN.....	6
2.5.2	MAX-phase materials	6
2.5.3	Indium tin oxide (ITO)	7
2.5.4	PEDT polymer	7
3	Sample manufacturing and characterization.....	8
3.1	Sample manufacturing.....	8
3.1.1	Physical vapour deposition (PVD).....	8
3.1.2	High temperature nitridation.....	8
3.2	Sample characterization	8
3.2.1	X-ray diffraction	8
3.2.2	Optical characterization with spectrophotometry	8
4	Results	10
4.1	Introduction	10
4.2	TiAlN	10
4.2.1	Measured reflectance of sputtered TiAlN (old)	11
4.2.2	Measured reflectance of sputtered TiAlN (new)	11
4.2.3	Measured reflectance of nitrided TiAl.....	12
4.3	MAX-phase materials.....	13
4.3.1	Simulations of thin film MAX-phase Ti_4AlN_3 and Ti_3Si_2	14
4.3.2	Measured reflectance of thin film MAX-phase Ti_3AlC_2 , Ti_3AlN_2 and Ti_2AlC	15
4.4	Simulated reflectance of sputtered indium tin oxide (ITO).....	16
4.5	Measured reflectance of a PEDT polymer	21
5	Summary and conclusions	22

1 Introduction

The instruments on the BepiColombo mission to Mercury (ESA, 2004) will be submitted to a harsh environment, receiving radiated power from the Sun and the planet about 10 orders of magnitude larger than that of an Earth bound orbit. The equipment that is not covered by the housing platform needs to be coated to protect it from overheating. The choice of coating material is not obvious, and this report presents measurements and simulations of some candidate materials.

The Swedish Institute of Space Physics (IRF) has previously sent Langmuir Probes with e.g. the Cassini/Huygens mission (called RPWS LP), and the Rosetta mission (called LAP). The TiN coatings used on these probes provide a worst-case balance temperature of 150 °C, calculated for the prevailing conditions. On BepiColombo, the Langmuir Probe will be a part of the MEFISTO sensor configuration (Blomberg et al., 2004) which also includes co-axial cables with heat sensitive insulation material.

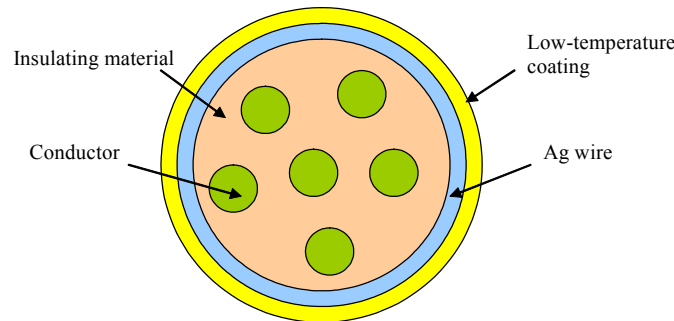


Fig. 1-1. Schematic cross-section of a co-axial boom cable with a low-temperature coating.

Temperature calculations of thin film TiN coatings has previously been reported (Karmhag and Ribbing, 1999), as well as thin film TiAlN (Brogren et al., 2000). TiN coatings manufactured by high temperature nitridation have also been investigated (Veszelei, 1997). The lowest balance temperature was found for the TiAlN coating, and calculations in a Mercury environment results in a balance temperature of 270 °C.

The aim of this study is to take advantage of the advanced instrumentation and highly qualified competence within the Ångström Laboratory and associated resources, in the search for a suitable coating for very hot space environments. For all the real and simulated materials, the purpose was to use the optical response and calculate the steady state balance temperature in a Mercury environment.

The balance temperature of a surface can be calculated from parameters obtained from optical characterization. Thin films of sputtered TiAlN were manufactured and optically analyzed. Thin films of sputtered TiAl were submitted to nitridation, and optical characterization of the films was performed. Previously obtained data (Hultåker, 2002) from optical characterization of thin films of indium tin oxide (ITO) were used to simulate the optical response at different film thicknesses. Previously obtained data (Pettersson, 2003) from optical characterization of bulk MAX-phase materials (this type of material is described in section 2.5.2) were used to simulate the optical response at different thicknesses. Thin films of MAX-phase materials were submitted to optical characterization. And the optical response of a PEDT polymer was obtained.

The candidate material for the probes was found to be a 0.65 µm sputtered $\text{Ti}_{0.16}\text{Al}_{0.43}\text{N}_{0.41}$ yielding a temperature of 270 °C ($\alpha/\epsilon = 1.3$). Simulations of sputtered ITO of 0.6 µm, onto Ag, yielded a temperature of 200 °C ($\alpha/\epsilon = 0.71$) which makes it an interesting material for the boom cables.

Participants in this project were:

Anna-Lena Larsson (Swedish Institute of Space Physics and Department of Engineering Sciences, Uppsala University), optical characterization, simulations and writing

Jan-Erik Wahlund (Swedish Institute of Space Physics), project driver

Urban Wiklund (Department of Engineering Sciences, Uppsala University), manufacturing of sputtered TiAl and TiAlN samples

Erik Johansson (Ti-Surf AB), manufacturing of nitrided TiAl samples

Annette Hultåker (Department of Engineering Sciences, Uppsala University), manufacturing and characterization of ITO samples

Lars Bylander (Alfvén Laboratory, The Royal Institute of Technology), project driver

Heléne Pettersson (Department of Engineering Sciences, Uppsala University), optical characterization of bulk MAX phase materials

Ola Wilhelmsson (Department of Materials Chemistry, Uppsala University), manufacturing of thin film Max phase materials

C-G Ribbing and Richard Karmhag (Department of Engineering Sciences, Uppsala University), discussion

Hans Högberg and Torbjörn Joelsson (IFM, Linköping University), manufacturing of thin film Max phase materials

2 Background

This chapter gives the physical background for the materials parameters searched for, the measurement techniques used, the requirements given by the parts to be coated and a short description of the candidate materials. Note that all formulas are written in SI units

2.1 Heat transfer in a Mercury environment

Heat can generally be transferred by conduction (within a solid or a non-moving liquid), convection (between a solid and liquid or gas) and radiation (between objects not in contact). Between the instrument and the surrounding space environment, heat is exchanged by radiation. Heat is led by conduction in the instrument structure, and exchanged by radiation between the internal surfaces. Convection will be neglected, as well as the internally dissipated heat. The radiation exchange with the environment gives rise to the largest contribution for the temperature changes of the MEFISTO sensor. The instrument will receive heat, q_{rec} , from the Sun and Mercury, and lose heat by thermal emittance, q_{em} , according to

$$\begin{aligned} q_{rec} &= \alpha AS + q_{Merc} \\ q_{em} &= \varepsilon A \sigma T^4 \end{aligned} ,$$

Eq. 1

where α is the solar absorptance (a material constant), A is the projected area, S is the solar constant (a function of wavelength) and q_{Merc} is the emitted radiation from the planet, ε is the emittance (a material constant), σ is the Stefan-Boltzmann constant, and T is the temperature of the emitting surface. The received radiation, including the albedo, in the vicinity of Mercury can be described by $q_{rec} = A \cdot S_0 \cdot I$ (JAXA, 2004). The material constants are described in section 2.2.1.

The equations can be submitted to a finite-element method to calculate the transient temperature variation, but this report focuses on the steady-state case for the worst-case scenario.

2.2 Optical properties of materials

2.2.1 Absorptance and emittance

The solar absorptance α , and the thermal emittance ε , in Eq. 1, are functions of wavelength and can be obtained from a material by measuring its reflectance, R . The spectral absorption is $A = 1 - R$, where A , R and T are functions of the wavelength, λ . The values of α and ε can be calculated by weighting the measured spectral absorption with the incoming solar radiation S (function of wavelength, λ) and the thermal blackbody spectrum, B (function of wavelength, λ , and temperature, T), and integrating over the particular wavelength interval (for a certain temperature). The weight function S used in this work is the solar intensity distribution in space (2000 ASTM Standard extraterrestrial spectrum reference E-490-00 at air mass 0) (NREL, 2000). The α and ε are thus obtained from

$$\alpha = \frac{\int_{0.3\mu m}^{2\mu m} d\lambda (1 - R_\lambda - T_\lambda) S_\lambda}{\int_{0.3\mu m}^{2\mu m} d\lambda S_\lambda} \quad e = \frac{\int_{2\mu m}^{50\mu m} d\lambda (1 - R_\lambda - T_\lambda) B_{T,\lambda}}{\int_{2\mu m}^{50\mu m} d\lambda B_{T,\lambda}},$$

Eq. 2

where λ is the wavelength, R_λ is the reflectance, T_λ is the transmittance, S_λ is the solar spectra and $B_{T,\lambda}$ is the black body radiation.

In this study, the emittance refers to the directional one, since it was obtained from Eq. 2 using the reflectance data for 25 degrees angle of incidence, for the infrared wavelength range. It is an approximation to the hemispherical emittance.

The optical constants can be obtained by different measurement techniques and inversion methods that fit models to the spectra. The reflectance (R) and transmittance (T) can be measured with spectrophotometry (see section 3.2.2), and submitted to Kramers-Kronig analysis. This method uses the coupling between the real and imaginary parts of the dielectric function. The optical response for all wavelengths is needed, so the method involves extrapolated values. This method was used to obtain the optical constants of the ITO (Hult aker, 2002) and the bulk MAX-phase materials (Pettersson, 2003).

2.2.2 Thin film interference

The absorption in a layer of thickness d decreases with $e^{-\alpha d}$, so the criterion for a film to be thin is that the optical density, αd , should be of the order of 1, or lower. Further, $\alpha \sim k/\lambda$, hence $kd/\lambda \leq 1$.

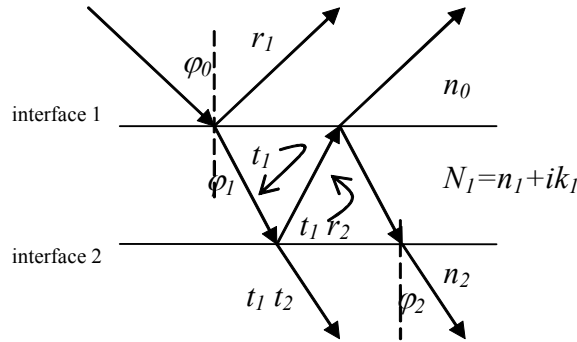


Fig. 2-1 Multiple reflections in a thin film layer of refractive index N_1 , the angle of incidence is φ_0 , the substrate refractive index is n_2 and the refractive index of the ambient medium is n_0

The thin film reflectance can be calculated with the Fresnel formalism (Born and Wolf, 1980) by

$$\delta = \frac{2\pi}{\lambda} N_1 d \cos \varphi_0$$

$$r_{1,2} = \frac{n_{0,1} - n_{1,2}}{n_{0,1} + n_{1,2}} \quad r = \frac{r_1 + r_1 r_2 e^{-2i\delta}}{1 + r_1 r_2 e^{-2i\delta}}$$

$$R = r \cdot r^*$$

Eq. 3

where δ is the phase, λ is the wavelength, N_1 is the complex refractive index of the film, d is the film thickness, φ_0 is the angle of incidence and $r_{1,2}$ are the reflectance coefficients, and R is the measurable intensity. The formulas in Eq. 3 are due for a single film on a semi-infinite substrate. The indices 1 and 2 correspond to the different interfaces shown in Fig. 2-1.

2.3 Environmental conditions

The MEFISTO orbit around Mercury takes 9.3 h and has 88 days of precession. The position and orientations of MEFISTO will also be affected by Mercury's orbit around the Sun, and the radiation received by the instrument can be summarized as follows.

- Radiation from Mercury, daytime including albedo: 7300-13600 W/m²
- Radiation from the Sun: 14500-6300 W/m²
- Worst-case hot scenario: The satellite is located between Mercury (+430 °C) and the sun.

2.4 Surface coatings requirements

The surface coating must

- be electrically conducting, and exhibit a uniform electrical work function that is free from induced EMF (to provide least possible hysteresis). The variance for the previously used TiN is 15 rms.
- be mechanically durable, e.g., be able to handle micro-meteorites, and also be easy to handle during manufacturing and integration.
- be chemically inert to atomic oxygen that prevails around most planetary bodies in space.
- be radiation resistant towards solar proton sputtering and cosmic rays.
- provide a balance temperature (for a specified substrate) near the Sun and Mercury that is as low as possible.

2.5 Materials selection

There are many surface coatings that would yield a low balance temperature, for example Al₂O₃, but the coating must be electrically conducting, which makes all ceramics (and other insulators) inappropriate. Metals are good conductors, but would in most cases yield a too low emittance number (due to the high reflectance at infrared wavelengths), which will give a high balance temperature. Ti nitride compounds (see section 2.5.1) seem to have optical properties between that of insulators and metals, and are suitable as low-temperature coatings. Indium tin oxide (ITO) has previously been used for space applications due to its conducting properties. It can be designed to keep the infrared reflectance at a moderate level, which makes it suitable as a low-temperature coating. A new class of highly ordered materials called MAX-phase materials (see section 2.5.2) are also interesting to consider due to their ceramic properties but yet good electrical conductivity. Another interesting group of materials are the conducting polymers.

2.5.1 Thin film TiN and TiAlN

Thin film coatings of TiN and TiAlN are widely used in the tools industry due to their good mechanical properties. They are also electrically conducting, and therefore suitable as coatings for probes used in electrical fields. The optical properties of thin films of TiN and TiAlN has previously been reported (Karmhag and Ribbing, 1999; Brogren et al., 2000). Calculations in an Earth-bound orbit resulted in temperatures of 110 and 34 °C respectively. The stoichiometry of the TiAlN film was Ti_{0.16}Al_{0.43}N_{0.41} (Brogren et al., 2000), and it was deposited onto Al.

Samples of TiAlN were manufactured from a Ti_{0.5}Al_{0.5} target by sputtering with nitrogen, and also by post-nitridation of sputtered TiAl films. Both types of films were deposited onto Ti substrates.

2.5.2 MAX-phase materials

The MAX-phase materials are highly ordered compounds that were “discovered” in the mid 90's (Barsoum and El-Raghy, 2001), where M is a transition metal, A is an element in group 13-14 of the periodic system, and X is carbon or nitrogen. For example, TiAlN can be manufactured as a MAX-phase. The materials can be described as ceramic materials with metallic properties. For example, Ti₃SiC₂ has a high thermal and electrical conductivity and exhibits one of the lowest friction coefficients of any solid measured to date. The compound has also a high stiffness combined with a low density and a very high

tolerance towards damage. Furthermore, it seems to maintain these properties at high temperatures, and is also highly resistant to oxidation and thermal shock.

The optical properties of thin films of MAX-phase materials have not been previously reported.

2.5.3 Indium tin oxide (ITO)

Indium tin oxide (ITO) (Granqvist and Hult aker, 2002) is widely known as a conductor transparent for visible light. It is frequently used in applications such as low-emissive windows and flat panel displays. Thin film ITO has been deposited onto instruments for space flight, to reduce charging, since ITO is a good conductor. It is also very hard, and has sometimes been used for mechanical protection. It is possible to manufacture ITO with a variety of optical properties, depending on the doping concentration.

2.5.4 PEDT polymer

Baytron[®] M is a monomer used in the manufacture of polymeric organic conductors. The conductive polymer formed by polymerization of Baytron[®] M is poly(3,4-ethylenedioxythiophene), or PEDT. PEDT/Tosylate made from Baytron[®] M can be used for the manufacture of intrinsically conductive yet highly transparent coatings (HCStarck, 2004). The manufacture of antistatic and/or conductive polymer coatings must be specifically adapted to the substrate to be coated.

3 Sample manufacturing and characterization

This chapter describes the techniques used for sample manufacturing and analysis.

3.1 Sample manufacturing

3.1.1 Physical vapour deposition (PVD)

Thin films can be manufactured by physical vapour deposition (PVD) such as sputtering and evaporation. Free atoms from a solid target are released for example by ion bombardment (sputtering) or heating (evaporation). The atoms can react and mix with other substances before or at the deposition on the substrate. Sputtering often involves higher energies of the sputtered compounds, and therefore a better adhesion in comparison to evaporation.

3.1.2 High temperature nitridation

In this process, the material is submitted to a nitrogenous atmosphere at an elevated temperature (ca 800 °C). The surface will react with the nitrogen and form a nitride. Since the nitridation proceeds by diffusion, there will be a gradient interface between the nitride and the material it is grown from.

3.2 Sample characterization

3.2.1 X-ray diffraction

X-ray diffraction (XRD) can be used to identify a material and give information about the structure of the material, e.g. phase, lattice stress, texture orientation and grain size (Cullity, 1956). It is based on the constructive interference between x-rays reflected at different atomic planes. By varying the angle between the incident and diffracted beam and recording the diffracted radiation, a chart with material and phase specific peaks as achieved. The films used in this work were submitted to grazing incidence X-ray diffraction, using a Siemens D5000-unit with CuK_α radiation of 1.54 Å.

3.2.2 Optical characterization with spectrophotometry

The reflectance measurements in the near-ultraviolet/visible/near-infrared range were done with a Perkin-Elmer Lambda 900 spectrophotometer. This is a single beam instrument operating in the range 300 to 2500 nm. The light is collected in an integrating sphere covered with Spektralon® which is also used as a reflectance reference.

The infrared optical measurements were recorded with a Perkin-Elmer 983 spectrophotometer. This is a double beam instrument with a rotating mirror operating in the range 2 to 50 µm. Sputtered gold mirrors were used as references, and the angle of incidence was 25°. The incident infrared radiation was to a good approximation equal to that of the unpolarized one. It was verified in several cases that the measured reflectance corresponded to the average of the values obtained for s- and p-polarized radiation. To improve the signal to noise ratio, an air dryer was used.

The area of the thin film MAX-phase samples was smaller than the incoming beam, so delimiting masks were used when measuring. The data correction was done according to

$$S_{sample} = \frac{S_{measured, sample+mask} - S_{measured, mask}}{S_{measured, reference} - S_{measured, mask}},$$

Eq. 4

where S_{sample} is the corrected signal from the sample, $S_{measured,sample+mask}$ is the signal from the sample with mask, $S_{measured,mask}$ is the signal from the mask only, $S_{measured,reference}$ is the signal from the reference measurement.

4 Results

This chapter gives a graphical presentation of the measured and simulated values.

4.1 Introduction

The reflectance is the key parameter when the balance temperature is to be determined. If the *measured reflectance* is inserted in Eq. 2 and Eq. 1, the balance temperature is obtained for the specific sample, at an arbitrary initial temperature. The calculated temperature is set to the new initial temperature, and the procedure is iterated until “equilibrium” is reached (i.e. the difference between the initial and resulting temperature is less than 0.5 degrees).

If the optical constants (see section 2.2.1) are known, the reflectance for different film thicknesses can be *simulated*, and an optimal thickness (yielding the lowest temperature) can be found. The temperature iteration for each thickness also remains. The optical constants for the substrate are needed as well.

In most of the graphs, the Gaussian shaped black body spectrum and the solar radiation spectrum are included (see section 2.2.1 about these weight functions). The blackbody radiation is due for *the coating* at the calculated “equilibrium” temperature. The calculated alfa, epsilon and temperature in °C (called TC) are also included. The x-axis shows the logarithmic wavelength in μm . All spectra are normalized to one.

4.2 TiAlN

The measured reflectance of the previously manufactured TiAlN film was used to calculate the balance temperature in a Mercury environment. It was found to be 270 °C (see Fig. 4-1), and the alfa/epsilon value was 1.3. The balance temperature for the new samples (see Fig. 4-2 to Fig. 4-8) varied between 300 and 400 °C, and the corresponding alfa/epsilon value varied between 1.6 and 3.0. The difference in reflectance for different film thicknesses was small. The sputtered films had lower balance temperatures than the nitrided ones. The sputtered TiAlN films in Fig. 4-1 (old type) and Fig. 4-3 (new type) were 0.65 and 0.60 μm thick, and both reflectance spectra exhibit a dip around the long wavelength edge of the black-body spectrum, which increases the emittance. No such feature was found in the nitrided films. The absorption of the sputtered TiAlN was quite high (≈ 0.75) while that of the nitrided films was considerably lower (≈ 0.42). The emittance of the nitrided films was unfortunately very low, yielding a high alfa/epsilon values.

4.2.1 Measured reflectance of sputtered TiAlN (old)

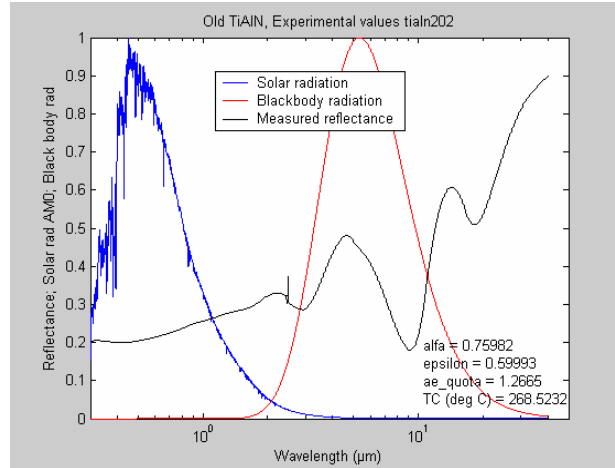


Fig. 4-1 Measured reflectance of previously sputtered TiAlN, 0.65 μm thick. The balance temperature was 270 $^{\circ}\text{C}$.

4.2.2 Measured reflectance of sputtered TiAlN (new)

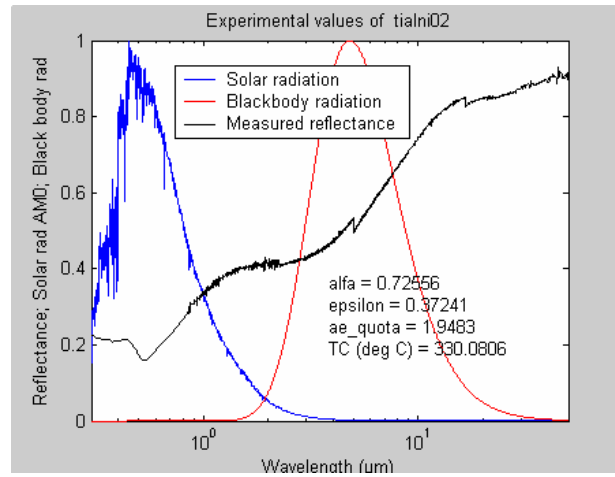


Fig. 4-2. Measured reflectance of sputtered TiAlN, 0.2 μm thick. The balance temperature was 330 $^{\circ}\text{C}$ and $\alpha/\epsilon = 1.9$.

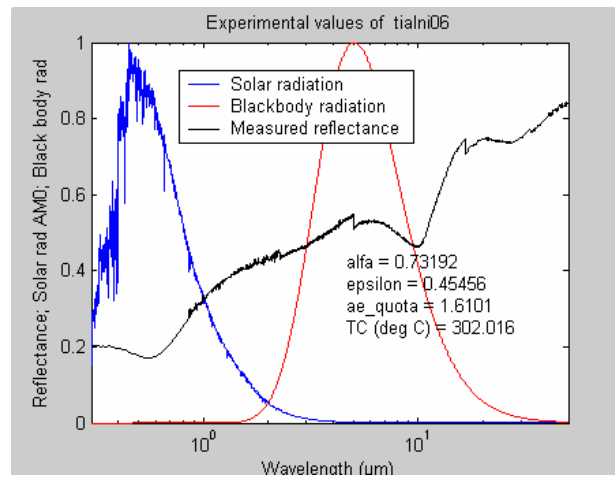


Fig. 4-3. Measured reflectance of sputtered TiAlN, 0.6 μm thick. The balance temperature was 300 $^{\circ}\text{C}$ and $\alpha/\epsilon = 1.6$.

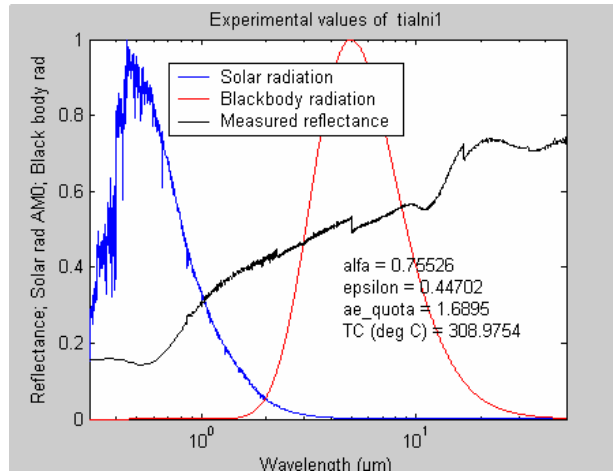


Fig. 4-4. Measured reflectance of sputtered TiAlN, 1.0 μm thick. The balance temperature was 310 $^{\circ}\text{C}$ and $\alpha/\epsilon = 1.7$.

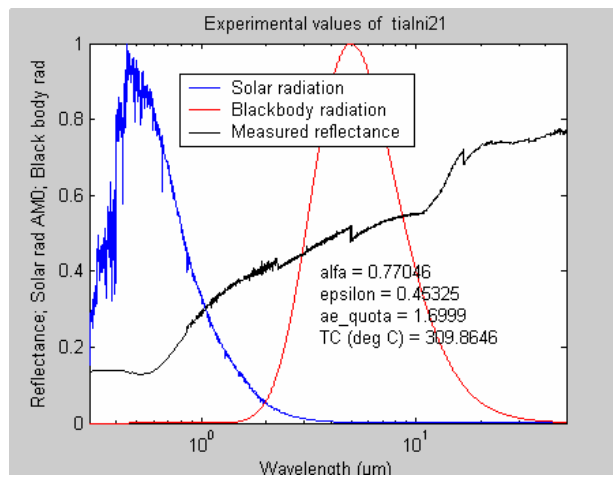


Fig. 4-5. Measured reflectance of sputtered TiAlN, 2.1 μm thick. The balance temperature was 310 $^{\circ}\text{C}$ and $\alpha/\epsilon = 1.7$.

4.2.3 Measured reflectance of nitrided TiAl

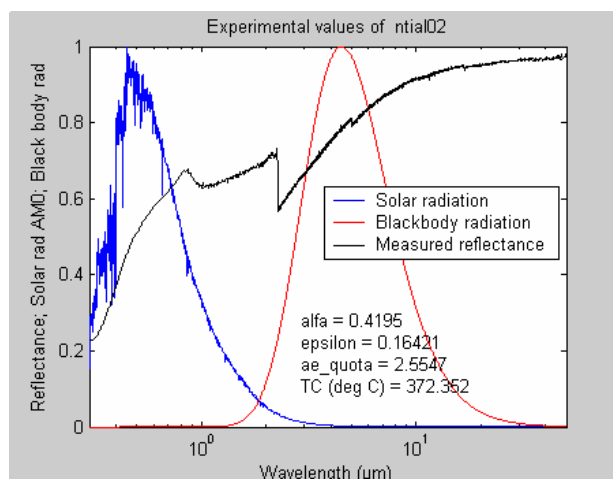


Fig. 4-6 Measured reflectance of nitrided TiAl, 0.2 μm thick. The balance temperature was 370 $^{\circ}\text{C}$ and $\alpha/\epsilon = 2.6$.

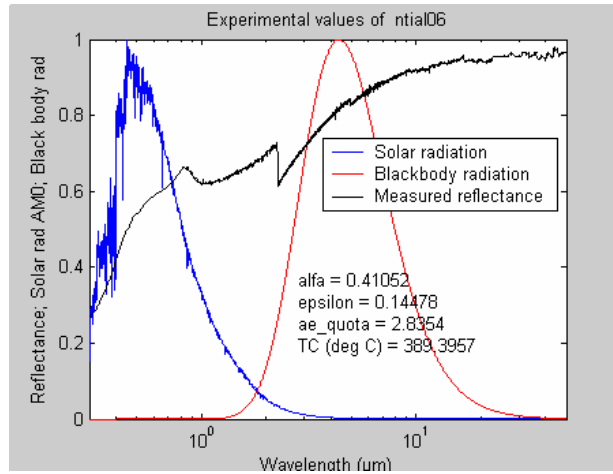


Fig. 4-7. Measured reflectance of nitrided TiAl, 0.6 μm thick. The balance temperature was 390 $^{\circ}\text{C}$ and $\alpha/\epsilon = 2.8$.

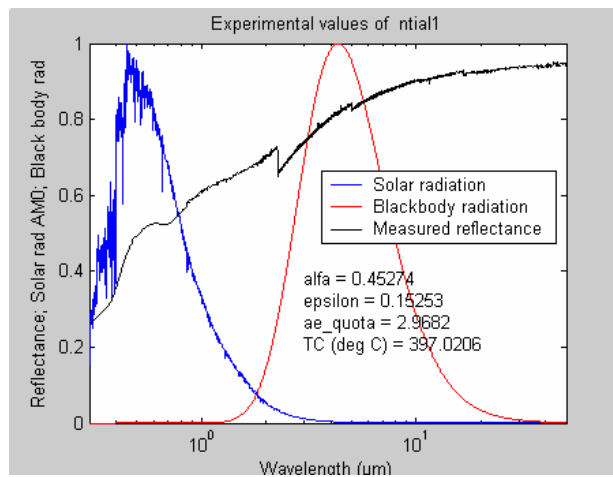


Fig. 4-8. Measured reflectance of nitrided TiAl, 1.0 μm thick. The balance temperature was 400 $^{\circ}\text{C}$ and $\alpha/\epsilon = 3.0$.

4.3 MAX-phase materials

The optical constants obtained from bulk Ti_4AlN_3 and Ti_3Si_2 were used to simulate the reflectance at different thicknesses. Fig. 4-10 and Fig. 4-11 shows the resulting balance temperature as a function of thickness. The simulated substrate was Ag. The optimal thicknesses were found to be 0.35 and 0.26 μm respectively. The corresponding balance temperatures were found to be 330 and 420 $^{\circ}\text{C}$, and the α/ϵ values were 1.9 and 3.3. The simulated thin film reflectance did not differ very much from the bulk reflectance (not shown here).

Thin films of MAX-phase Ti_3AlC_2 , Ti_3AlN_2 and Ti_2AlC were grown on TiC to a thickness of 0.2 μm . (The TiC substrate is used to get the highly ordered structure.) They were submitted to reflectance measurements and the balance temperature was calculated to 460, 460 and 490 $^{\circ}\text{C}$. The corresponding α/ϵ values were 4.2, 3.0 and 5.1. The reflectance did not vary considerably between the different films. The absorption of these films was moderate, but the emittance was too low.

4.3.1 Simulations of thin film MAX-phase Ti_4AlN_3 and Ti_3Si_2

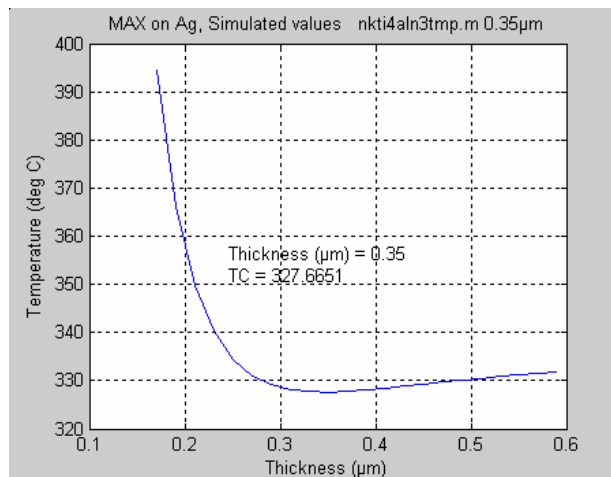


Fig. 4-9. Iteration of calculation of balance temperature for different thickness of Ti_4AlN_3 (on Ag). The lowest temperature was 330 °C for 0.35 μm .

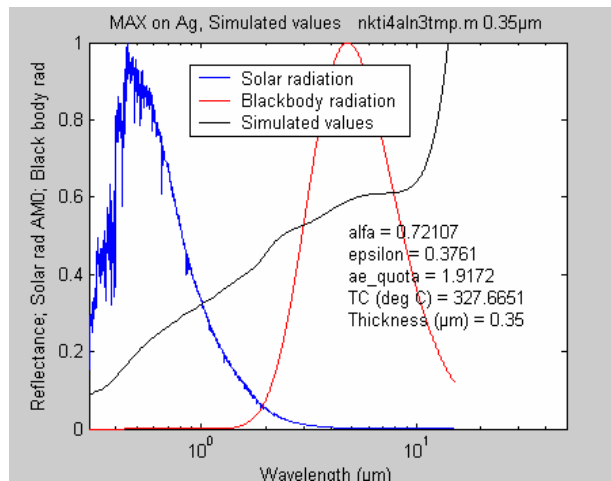


Fig. 4-10. Simulated reflectance of Ti_4AlN_3 (on Ag) at a thickness of 0.35 μm , yielding a balance temperature of 330 °C and $\alpha/\epsilon = 1.9$.

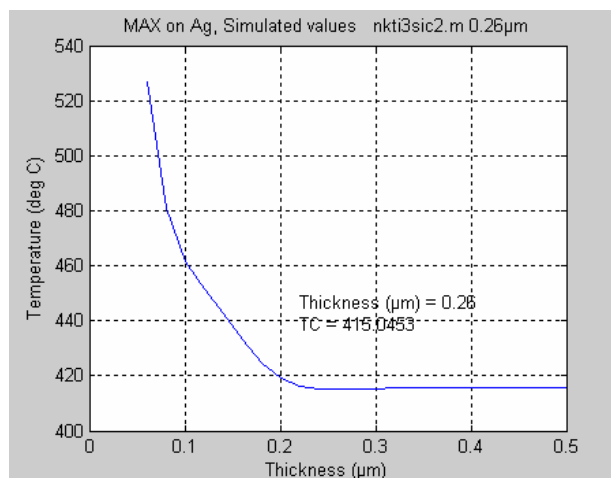


Fig. 4-11. Iteration of calculation of balance temperature for different thickness of Ti_3SiC_2 (on Ag). The lowest temperature was 420 °C for 0.26 μm .

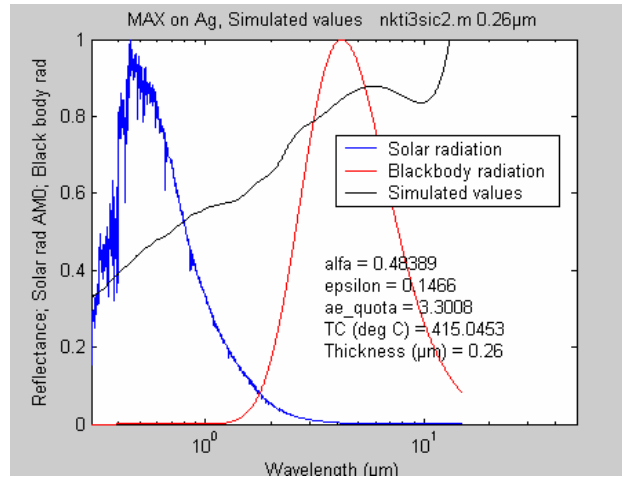


Fig. 4-12. Simulated reflectance of Ti_3SiC_2 (on Ag) at a thickness of $0.26 \mu\text{m}$, yielding a balance temperature of 420°C and $\alpha/\epsilon = 3.3$.

4.3.2 Measured reflectance of thin film MAX-phase Ti_3AlC_2 , Ti_3AlN_2 and Ti_2AlC

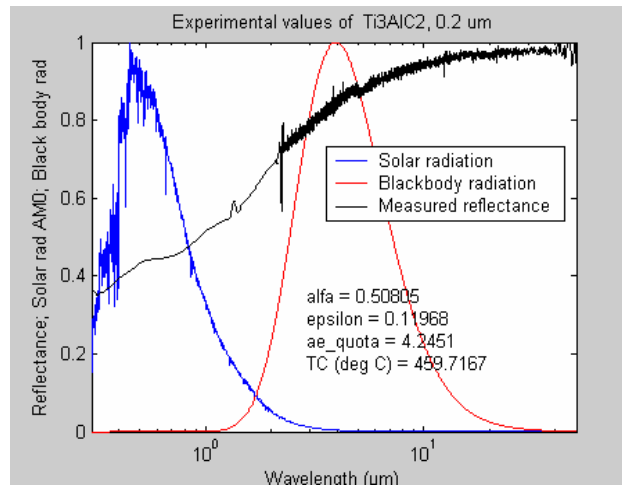


Fig. 4-13. Measured reflectance of thin film Ti_3AlC_2 , $0.2 \mu\text{m}$ thick. The balance temperature was 460°C and $\alpha/\epsilon = 4.2$.

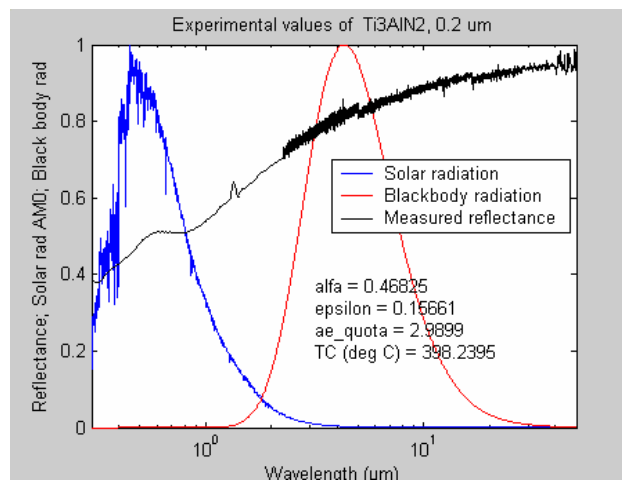


Fig. 4-14. Measured reflectance of thin film Ti_3AlN_2 , $0.2 \mu\text{m}$ thick. The balance temperature was 460°C and $\alpha/\epsilon = 3.0$.

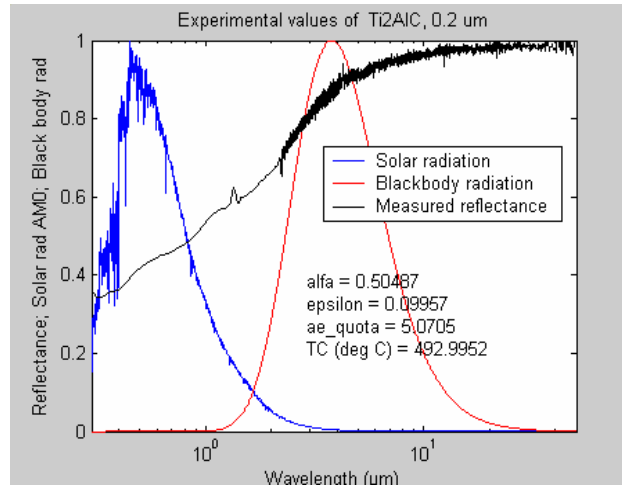


Fig. 4-15. Measured reflectance of thin film Ti_2AlC , 0.2 μm thick. The balance temperature was 490 °C and $\alpha/\epsilon = 5.1$.

4.4 Simulated reflectance of sputtered indium tin oxide (ITO)

Thin films of ITO were previously manufactured and optically analyzed in the thesis of Annette Hultåker (Hultåker, 2002). Page 67 of the thesis is reprinted in Fig. 4-15. It shows the spectral reflectance and transmittance of ITO samples with a moderately low infrared reflectance and a high transmittance at shorter wavelengths, which should yield a low α/ϵ value (see section 2.2.1). All films were deposited onto Corning glass 7059, and the thickness was around 120 nm. The optical constants were obtained from Kramers-Kronig analysis (Hultåker, 2004). Simulations of the reflectance of the ITO onto Corning 7059 glass are compared to the measured values (in Fig. 4-16, Fig. 4-19, Fig. 4-22 and Fig. 4-25) to verify the validity of the optical constants. The thicknesses were found to be around 120 μm , in agreement with the real films. The simulated reflectance is erroneous at long wavelengths, but this is of less importance since the black body radiation is approximately zero in this region.

A selection of interesting ITO films was simulated with an Ag substrate, for different film thickness. The lowest balance temperature, 200 °C, was found for the film corresponding to batch D n.a. (“not annealed”) in Fig. 4-15, for a thickness of 0.6 μm .

The simulated reflectance for ITO on glass in Fig. 4-20 and Fig. 4-23 is lower than the measured one, where the blackbody spectrum is large, and the emittance is probably slightly overestimated. This is not the case for the film in Fig. 4-17, which yielded the lowest balance temperature.

The results are due for a flat sample, while the real geometry will be a twinned plait.

There is a difference between the two samples that were deposited at room temperature and not heat-treated. The reasons for this might be: (1) because of the different oxygen partial pressure during the sputtering or (2) because of some chemical variations originating from the hysteresis behaviour of the target surface (see section 3.1.2). The thicknesses of the samples are similar, 120 to 130 nm and can therefore not be the cause of the differences.

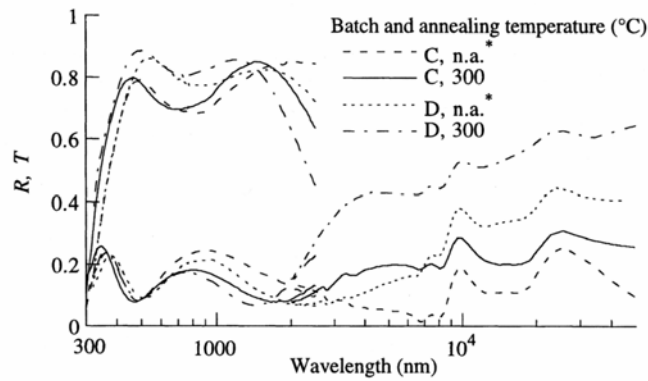


Figure 32: Spectral reflectance (R) and transmittance (T) for pure ITO samples from batches C and D, all deposited at room temperature and two were annealed at 300°C (T_A).

* n.a. means no annealing.

Tab. 5.25

67

Fig. 4-16 Page 67 from the thesis of Annette Hultåker (Hultåker, 2002), showing the measured reflectance and transmittance for different ITO samples for different annealing times.

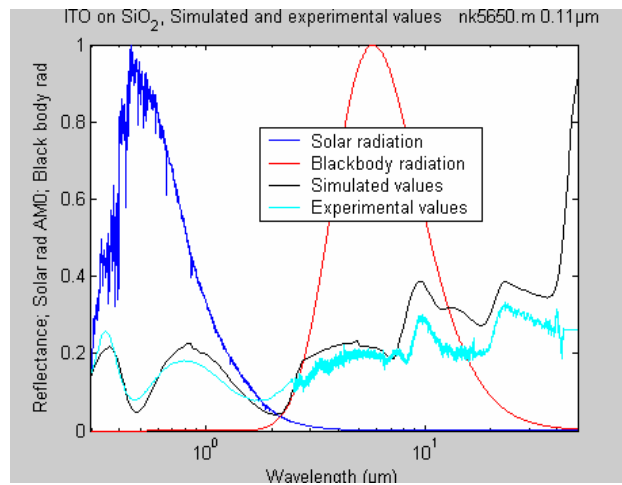


Fig. 4-17. Simulated and measured reflectance of ITO (type 5650) onto Corning 7059 glass.

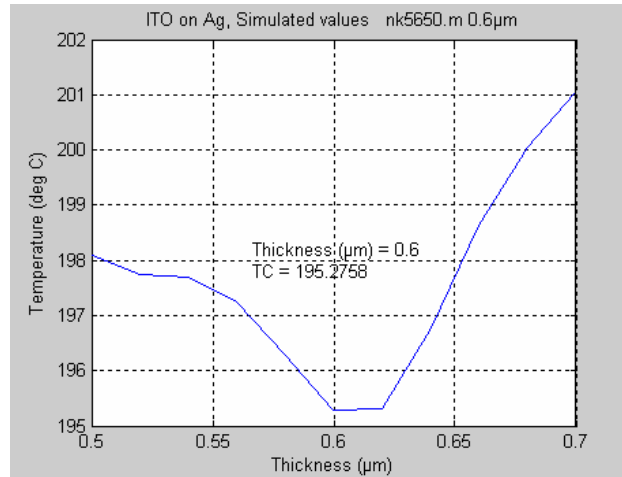


Fig. 4-18. Iteration of balance temperature calculations for different thickness of ITO (type 5650) on an Ag substrate. The lowest temperature was 200 °C for 0.6 µm.

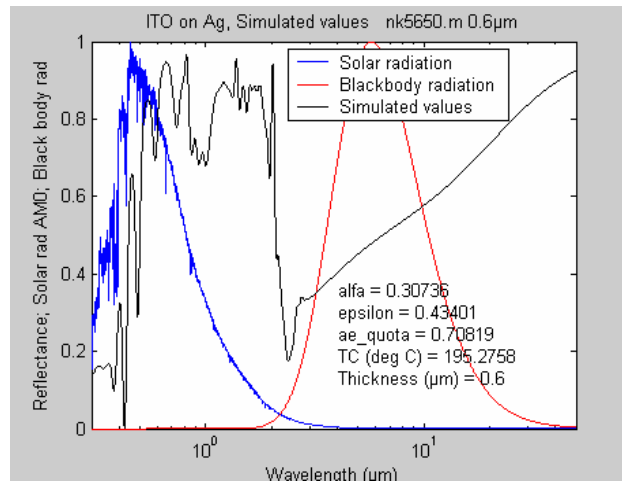


Fig. 4-19. Simulated reflectance of 0.6 µm ITO (type 5650) on an Ag substrate. The balance temperature was 200 °C and alfa/epsilon = 0.71.

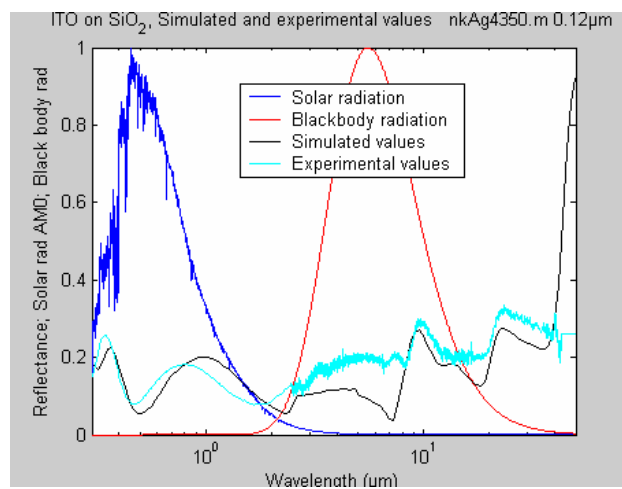


Fig. 4-20. Simulated and measured reflectance of ITO (type 4350) onto Corning 7059 glass.

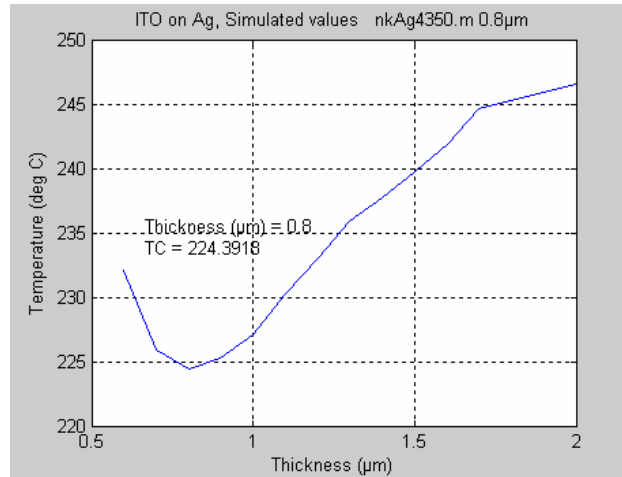


Fig. 4-21. Iteration of balance temperature calculations for different thickness of ITO (type 4350) on an Ag substrate. The lowest temperature was 220 °C for 0.8 μm.

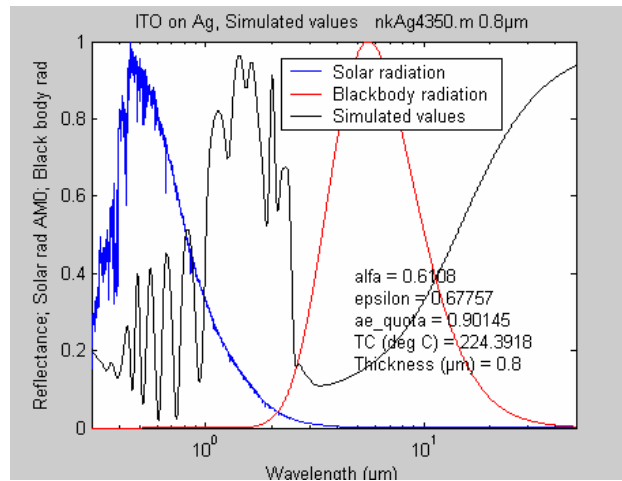


Fig. 4-22. Simulated reflectance of 0.8 μm ITO (type 4350) on an Ag substrate. The balance temperature was 220 °C and alfa/epsilon = 0.9.

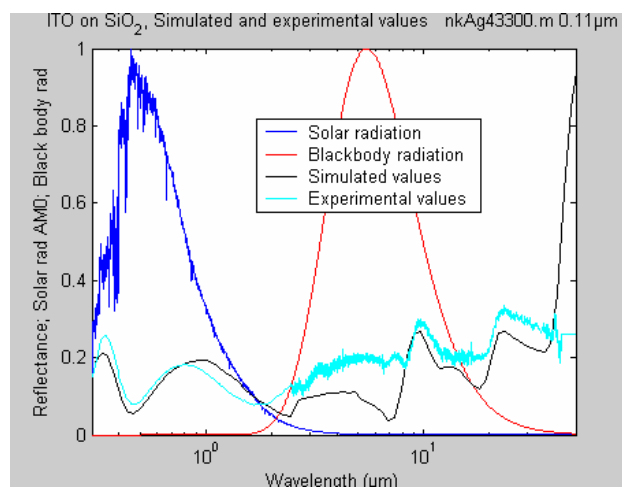


Fig. 4-23. Simulated and measured reflectance of ITO (type 43300) onto Corning 7059 glass.

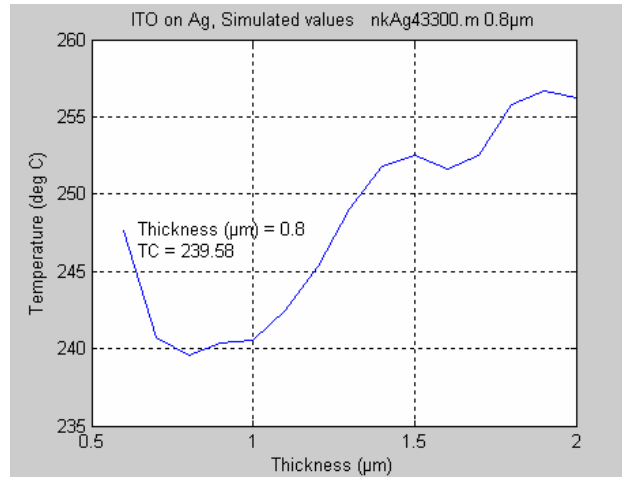


Fig. 4-24. Iteration of balance temperature calculations for different thickness of ITO (type 43300) on an Ag substrate. The lowest temperature was 240 °C for 0.8 μm

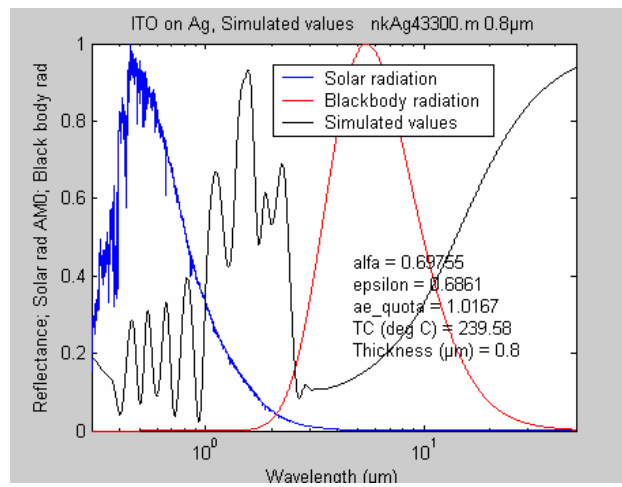


Fig. 4-25. . Simulated reflectance of 0.8 μm ITO (type 43300) on an Ag substrate. The balance temperature was 240 °C and $\alpha/\epsilon = 1.0$.

4.5 Measured reflectance of a PEDT polymer

The PEDT polymer was deposited onto a polished silver substrate. The thickness was of several μm , so it is not considered as a thin film. The balance temperature was found to be 280°C and the α/ϵ value was 1.4.

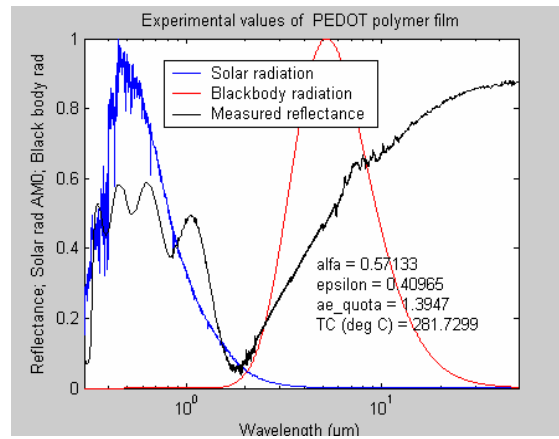


Fig. 4-26. Measured reflectance of a PEDT polymer onto Ag. The balance temperature was 280°C and $\alpha/\epsilon = 1.4$.

5 Summary and conclusions

The balance temperature, with the environmental conditions of Mercury, has been calculated from

- the measured reflectance of previously manufactured thin films of TiAlN,
- the measured reflectance measured from sputtered thin films of TiAlN and nitrided TiAl,
- the measured reflectance of thin films of MAX-phase compounds,
- the simulated reflectance obtained from optical constants of bulk MAX-phase compounds,
- the simulated reflectance obtained from optical constants of thin film ITO and
- the measured reflectance of a PEDT polymer.

The lowest balance temperature from measured reflectance was found for the previously manufactured TiAlN, $T = 270\text{ }^{\circ}\text{C}$ and $\alpha/\epsilon = 1.3$. The lowest temperature for simulated values was obtained from the ITO, $T = 200\text{ }^{\circ}\text{C}$ and $\alpha/\epsilon = 0.71$.

There was no decrease in temperature with the new types of TiAlN. These samples were manufactured from a target with a fixed composition (50% Ti and 50% Al), while the previously reported film had the stoichiometry $\text{Ti}_{0.16}\text{Al}_{0.43}\text{N}_{0.41}$. The MAX phase materials yielded temperatures between 330 and 490 $^{\circ}\text{C}$, which make them inappropriate for this type of application. The PEDT polymer showed a quite low balance temperature of 280 $^{\circ}\text{C}$, but the most interesting results came from the simulations of the ITO films. The lowest balance temperature of the ITO films investigated was 200 $^{\circ}\text{C}$. The ITO film can be designed to yield different optical properties, and it is probably possible to further optimize it for a low balance temperature, since this was not the aim of the previous work.

Conclusions: The results indicate that a 0.65 μm sputtered $\text{Ti}_{0.16}\text{Al}_{0.43}\text{N}_{0.41}$ is a candidate material for the probes, yielding a balance temperature of less than 300 $^{\circ}\text{C}$.

Simulations showed that 0.6 μm film of ITO of batch “D n.a.” (Hultåker, 2002), onto Ag, yielded 200 $^{\circ}\text{C}$ for a flat sample configuration. The ITO is considered to be a candidate material for the co-axial cables.

References

Barsoum, M. W. and T. El-Raghy (2001). "The MAX phases: Unique new carbide and nitride materials." *American Scientist* **89**: 334-343.

Blomberg, L. G., H. Matsumoto, J.-L. Bougeret, H. Kojima, S. Yagitani, M. André, L. Bylander, J. A. Cumnock, A. I. Eriksson, P.-A. Lindqvist, G. T. Marklund, M. Morooka, J. E. Wahlund, L. Åhlén, J. A. Holtet, E. Kallio, Y. Kasaba, K. Lappalainen, B. Lybekk, M. Moncuquet, K. Mursula, A. Mälkki, H. J. Opgenoorth and J. A. Trotignon, MEFISTO - An electric field instrument for BepiColombo/MMO, *Adv. Space Res.*, (*to be submitted*).

Born, M. and E. Wolf (1980). *Principles of optics*. London, Pergamon Press.

Brogren, M., G. L. Harding, R. Karmhag, C. G. Ribbing, G. A. Niklasson and L. Stenmark (2000). "Titanium-aluminum-nitride coatings for satellite temperature control." *Thin Solid Films* **370**(1): 268-277.

Cullity, B. D. (1956). *Elements of x-ray diffraction*. Massachusetts, Addison-Wesley.

ESA, http://www.esa.int/export/esaSC/120391_index_0_m.html, 2004

Granqvist, C. G. and A. Hultåker (2002). "Transparent and conducting ITO films: New developments and applications." *Thin Solid Films* **411**(1): 1-5.

HCStarck, <http://www.hcstarck-chemicals.com/>, 2004

Hultåker, A. Transparent conductive tin doped indium oxide. Ph.D. thesis, (Dep. of Materials Science, Uppsala University: 138, 2002).

Hultåker, A., Personal communication, 2004

JAXA, Personal communication, 2004

Karmhag, R. and C. G. Ribbing (1999). "TiN-alloy coatings for temperature control of space vessels." *Applied Optics* **38**(4): 674-678.

NREL, <http://rredc.nrel.gov/solar/spectra/am0/ASTM2000.html>, 2000

Pettersson, H. Optical characterization of two MAX-compounds. Master's thesis, Dep. of Materials Science, 2003).

Veszelei, M. Preparation and characterization of sputtered metal oxides and nitrides for optical applications. Ph.D. Thesis, (Dep. of Materials Science, Uppsala University, 1997).