

Studies on Tribological Behavior of Aluminum Nitride-Coated Steel*

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Abstract. The new opportunities introduced by the large development of the IoT (internet of things) are increasing the demand for sensors to be located as close as possible to the supervised process. The Aluminum Nitride (AlN) is one of the most promising materials for sensors due to its piezoelectric, excellent mechanical properties, chemical inertness and high melting point. Due to these material properties, the AlN sensors are suitable to operate in high temperature and harsh environment conditions and therefore are very promising to be employed in industrial applications. In this article are presented the studies conducted on several Aluminum Nitride-Coated Steel structures with the goal of producing sensors embedded in the ball bearings, bearings and other mobile parts of machine tools. The experiments were conducted on simple coatings structures without lubricating materials and the obtained results are promising, demonstrating that, with some limitations the AlN could be used in such applications.

1. Introduction

The Industrial Internet of Things (IIoT), could be considered as an extension of automation and connectivity also known as machine-to-machine (M2M) communication. IIoT is, growing in parallel with Internet of Things (IoT) due to the spectacular development of smart and connected [1].

People working in industrial environment would like to have smart sensors, with low energy consumption - eventually capable of harvesting energy from the machine tool where is installed, capable to withstand harsh environmental condition such as temperature, abrasion etc, wireless connected with the machine automation system and with factory's monitoring system.

Such a multitude of requirements are fulfilled only by some special materials, consequently it is necessary to investigate which are the most suitable and furthermore which could be produced through techniques adequate for the desired applications.

A basic criterion in choosing the material will be its possibility of being used for developing energy harvesting devices, consequently piezoelectric properties of material will be a must. Another criterion will be its possibility to be embedded on the monitored component of the machine tool. Although there it is a large area of piezoelectric materials there are limitations in regard with their capability of working in harsh industrial environment and not the last technical possibility of using them as presented in table 1.

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Table 1. Comparison of major characteristics of piezoelectric materials [1].

material/ properties	Pb(Zr,Ti)O ₃	SiO ₂	GaPO ₄	La ₂ Ga ₂ SiO ₁₄	AlN	LiNbO ₃
usable temperature (C ⁰)	250	350	920	1000	1200	1200
piezoelectric constant d ₃₃ (pC/N)	6	2	6	6	6	35
Available technology	Yes	No	No	No	Yes	Yes

As it could be observed in the above listed table the choice is somehow limited in this area. Although AlN (Aluminum Nitride) is a well known material, extensively studied for sensing applications in the last 20 years [2,3], for industrial applications its applications are not exhausted. AlN piezoelectric layers could be deposited by sputtering directly on the equipment parts needed to be monitored. Therefore it is possible to obtain embedded sensors, avoiding the reliability problems associated with the bonding or sensor attachment. For applications on which we are interested in (sensors applied on the surface of the element to be monitored) radio-frequency magnetron sputtering (RF-MS) is the only technical possibility. The deposition of highly structured AlN is dependent on the underlying films, the topography of the surface, and the deposition process parameters [4,5,6,7].

2. Sensor element manufacturing

With the goal of testing mechanical properties of the AlN layer deposited on materials similar to the one used in the machine tools, bearings, and ball bearings two samples with 60X60 mm dimensions were manufactured as presented in figure 1. It was decided to manufacture, one sample on Stainless Steel S3400 substrate and one sample on Inconel substrate. The reason to use two different substrates was also to test the different thermal behavior of AlN layer on these two materials.

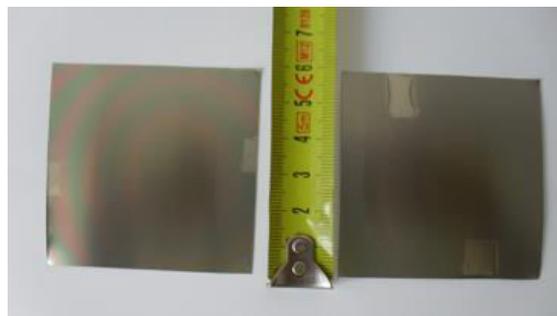


Figure 1. The two samples produced (Inconel left, Stainless Steel 3400 right).

Highly oriented (with *c* axis perpendicular on substrate) wurtzitic aluminum nitride (AlN) thin films were deposited by reactive radio-frequency magnetron sputtering (RF-MS) on stainless steel (SS) foil for sensor element fabrication. This type of substrate has few advantages: (i) it is flexible, and therefore the obtained sensor could be conformally attached to some mechanical parts that need to be monitored having also non-planar forms; (ii) it is resistant to the high temperatures that eventually could be developed in some situations during vibration monitoring; (iii) opens the way of realizing in the future the piezoelectric vibration sensor by depositing directly the AlN layer on the steel pieces that need to be monitored.

A high purity aluminum target (99.9999%, Mateck GmbH) with thickness of 2 mm and diameter of 110 mm was used for depositing the AlN films. The target was fixed on a magnetron cathod whose plasma ring is about 55 mm. Before their introduction into the deposition chamber, the substrates were

successively cleaned ultrasonically in acetone and isopropyl alcohol for 10 min and then dried in argon flow. The target-to-substrate separation distance was set at 35 mm.

Firstly the deposition chamber was evacuated down to a base pressure of $\sim 2 \times 10^{-4}$ Pa, and then the high purity working gases – argon and nitrogen – were admitted. The selected working pressure was 0.2 Pa and nitrogen partial pressure was 25%. The total gas flow was 40 sccm. The gas flows were controlled with Teledyne Hasting electronic mass flow meters and the gas pressure was monitored by an Alcatel capacitance vacuum gauge.

Prior to the deposition, the target was sputter-cleaned for 20 min in argon and then another 10 min in the working atmosphere (argon nitrogen mix). During target cleaning, the substrates were masked with a stainless steel shield in order to avoid undesired depositions onto them. Further on, the substrates were etched for 15 min in argon plasma produced by a wolfram plasmatron, in order to remove thin native oxide layers or other impurities which might persist after the ultrasonic cleaning.

An AlN layer with thickness of about 1 micron was then deposited on the stainless steel substrate with a deposition rate of about $14 \text{ nm} \cdot \text{min}^{-1}$ using a radio-frequency (1.78 MHz) generator and maintaining a low RF power ($\sim 100 \text{ W}$) in order to avoid overheating of the target surface. The substrates were not heated during deposition and their temperature was only dependent on plasma self-heating ($\sim 50\text{C}^0$) [6].

By using the same approach an AlN layer of about 1 micron was deposited on Inconel substrate.

The crystallographic quality of the AlN layers was investigated by X-ray diffraction (XRD) using a Bruker-AXS D8 Advance diffractometer in parallel beam setting, with $\text{CuK}\alpha$ radiation. Measurements were performed in the typical symmetric (θ - θ) geometry and by rocking curves (RC) in order to determine the degree of angular dispersion of the c-axis of the AlN crystallites around the surface normal.

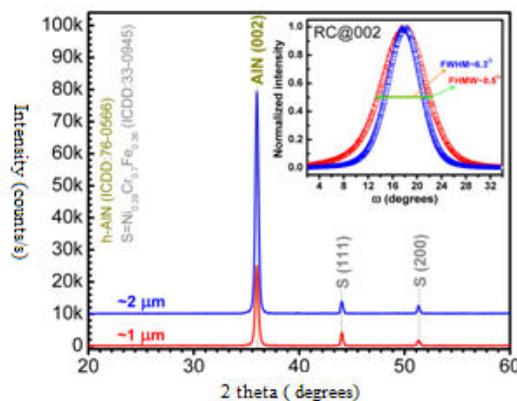


Figure 2. Symmetric geometry XRD patterns of AlN/Inconel sputtered films. Insert: rocking curves corresponding to the (002) AlN crystal planes.

The XRD patterns (figure 2), indicated that AlN films deposited on Inconel type substrate elicit only the 002 diffraction line of the hexagonal AlN phase (ICDD: 76-0566). This is indicative of a strongly c-axis textured material. The diffraction maxima of the metallic substrate have also been observed due to the low thickness of the sputtered films.

The full width at half maximum (FWHM) of the RCs, associated to the standard deviation of the crystallites' alignment, decreases from 8.6° to 6.3° (figure 2-insert), when the AlN film thickness is increased from $\sim 1 \mu\text{m}$ to $\sim 2 \mu\text{m}$, suggestive a gradual orientation of crystallites during film growth.

3. Tests conducted

The programme of testing the AlN layers was developed based on the challenges predicted to be encountered by the sensing elements to be developed. Thus it was established that only materials which could be coated with AlN and may withstand high temperatures (above 200C^0) would be further tested for the other requirements.

3.1. Temperature testing

The testing of samples behavior on different temperature were conducted by using a thermal chamber CTS - Thermal Shock Test Chambers, Series TSS capable of delivering temperatures up to 600C⁰ presented in figure 3.

According to the technical literature the following data for the temperature tests were considered: Thermal expansion coefficient: AlN: $4.15 \times 10^{-6} \text{ K}^{-1}$ [7] Stainless steel S 3400: $17.3 \times 10^{-6} \text{ K}^{-1}$ [8], Inconel: $11.5 \times 10^{-6} \text{ K}^{-1}$ [9].

It was interesting to observe that after exposing the sample with base material stainless steel to a temperature around 200C⁰ on the sample occurred exfoliations of the AlN layer (figure 4). For the sample with base material of Inconel the temperature raised up to 500C⁰ without occurring any exfoliations. Thus the decision taken was to continue the tests only on the samples which had been manufactured on Inconel base material.



Figure 3. CTS Thermal Shock Test Chambers, Series TSS.



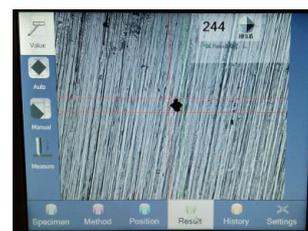
Figure 4. Exfoliation occurred on stainless steel sample at 200C⁰.

3.2. Hardness

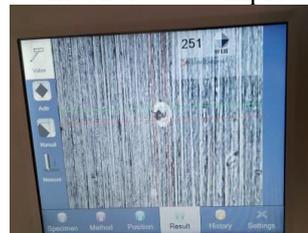
The test equipment used for hardness tests was the Dura Scan – Emco TEST Micro hardness tester with extended load range from 10 gf - 10 kgf for Vickers and Knoop tests. This system presents a unique system of combined closed- loop and dead weight technology ensuring accurate and repeatable test loads. In addition the completely automatic test cycles eliminate operator influence. The test machine is presented in figure 5 and the test results in figure 6.



Figure 5. Emco TEST Micro hardness tester.



Inconel base sample



Stainless Steel S3400 base sample

Figure 6. The two test results for Inconel and Stainless steel bases.

3.3. Abrasion testing

Two Inconel coated with AlN samples were tested, using different testing parameters as presented in figure 7, on CSM Instruments tribometer (figure 8). The block diagram of tribometer active section is presented in figure 11. In addition in order to have a reference two samples of Inconel without coating were tested by using similar parameters.

AlN sample 2-v0.1			
Tribo parameters			
Acquisition Radius : 6.01 [mm] Lin. speed : 0.10 [m/s] Normal load : 1.00 [N] Stop condit. : 50.00 [m] Effective stop : meters Acquisition rate : 2.6 [hz]	Sample -Coating : AlN -Substrate : INCONEL -Cleaning : MEC -Supplier : INOE	Static partner -Coating : No -Substrate : 100Cr6 -Cleaning : M.E.C. -Supplier : CSM -Dimension : 6.00 [mm] -Geometry : Ball	Environment Temperature : 22.50 [°C] Atmosphere : Air Humidity : 56.60 [%]
Sample Worn track section : 403.7 μm^2 Young's modulus : 310.0 gpa Poisson ratio : 0.290	Static partner Worn cap diameter : 244.0 μm Young's modulus : 200.0 gpa Poisson ratio : 0.300	Calculations Sample wear rate : 0.0003049 mm ³ /n/m Partner wear rate : 1.16E-006 mm ³ /n/m Max Herzian stress : 0.7254 gpa	
AlN sample 2-v0.2			
Tribo parameters			
Acquisition Radius : 7.06 [mm] Lin. speed : 0.20 [m/s] Normal load : 1.00 [N] Stop condit. : 50.00 [m] Effective stop : meters Acquisition rate : 4.5 [hz]	Sample -Coating : AlN -Substrate : INCONEL -Cleaning : MEC -Supplier : INOE	Static partner -Coating : No -Substrate : 100Cr6 -Cleaning : M.E.C. -Supplier : CSM -Dimension : 6.00 [mm] -Geometry : Ball	Environment Temperature : 22.50 [°C] Atmosphere : Air Humidity : 56.60 [%]
Sample Worn track section : 173.1 μm^2 Young's modulus : 310.0 gpa Poisson ratio : 0.290	Static partner Worn cap diameter : 184.0 μm Young's modulus : 200.0 gpa Poisson ratio : 0.300	Calculations Sample wear rate : 0.0001536 mm ³ /n/m Partner wear rate : 3.751E-007 mm ³ /n/m Max Herzian stress : 0.7254 gpa	
INCONEL sample 2-v-01			
Tribo parameters			
Acquisition Radius : 6.01 [mm] Lin. speed : 0.10 [m/s] Normal load : 1.00 [N] Stop condit. : 50.00 [m] Effective stop : meters Acquisition rate : 2.6 [hz]	Sample -Coating : No -Substrate : INCONEL -Cleaning : MEC -Supplier : INOE	Static partner -Coating : No -Substrate : 100Cr6 -Cleaning : M.E.C. -Supplier : CSM -Dimension : 6.00 [mm] -Geometry : Ball	Environment Temperature : 22.50 [°C] Atmosphere : Air Humidity : 56.60 [%]
Sample Worn track section : 39.0 μm^2 Young's modulus : 210.0 gpa Poisson ratio : 0.300	Static partner Worn cap diameter : 152.0 μm Young's modulus : 200.0 gpa Poisson ratio : 0.300	Calculations Sample wear rate : 2.946E-005 mm ³ /n/m Partner wear rate : 1.746E-007 mm ³ /n/m Max Herzian stress : 0.6483 gpa	
INCONEL sample 2-v0.2			
Tribo parameters			
Acquisition Radius : 7.03 [mm] Lin. speed : 0.20 [m/s] Normal load : 1.00 [N] Stop condit. : 50.00 [m] Effective stop : meters Acquisition rate : 4.5 [hz]	Sample -Coating : No -Substrate : INCONEL -Cleaning : MEC -Supplier : INOE	Static partner -Coating : No -Substrate : 100Cr6 -Cleaning : M.E.C. -Supplier : CSM -Dimension : 6.00 [mm] -Geometry : Ball	Environment Temperature : 22.50 [°C] Atmosphere : Air Humidity : 56.60 [%]
Sample Worn track section : 83.0 μm^2 Young's modulus : 210.0 gpa Poisson ratio : 0.300	Static partner Worn cap diameter : 192.0 μm Young's modulus : 200.0 gpa Poisson ratio : 0.300	Calculations Sample wear rate : 7.329E-005 mm ³ /n/m Partner wear rate : 4.449E-007 mm ³ /n/m Max Herzian stress : 0.6483 gpa	

Figure 7. The testing parameters.

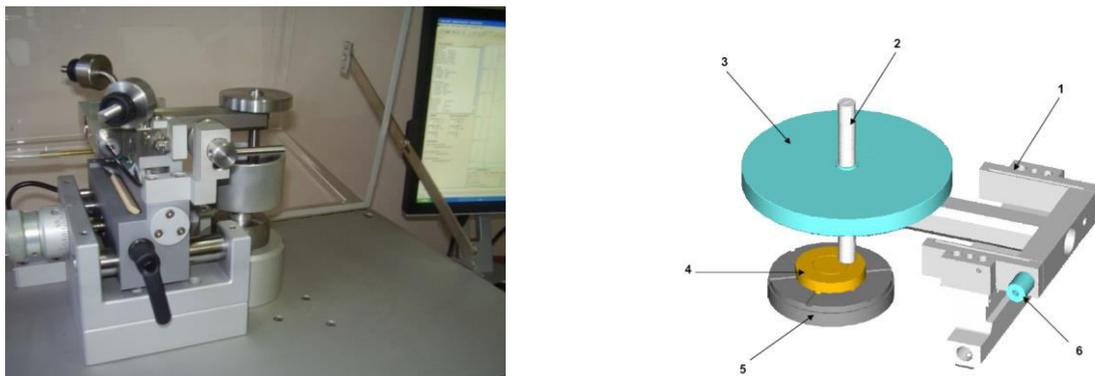


Figure 8. The CSM Instruments tribometer (a), the block diagram of tribometer active section (b):
 1. Elastic shim; 2. Ball fixture; 3. Weights; 4. Fixture for sample; 5. Tangential force sensor.

The obtained results are presented in figure 9 and 10.

Samples were also observed using the electronic microscope Hitachi and the results are presented in figure 11 and figure 12, where it could be observed that the AlN layer is eroded quite fast.

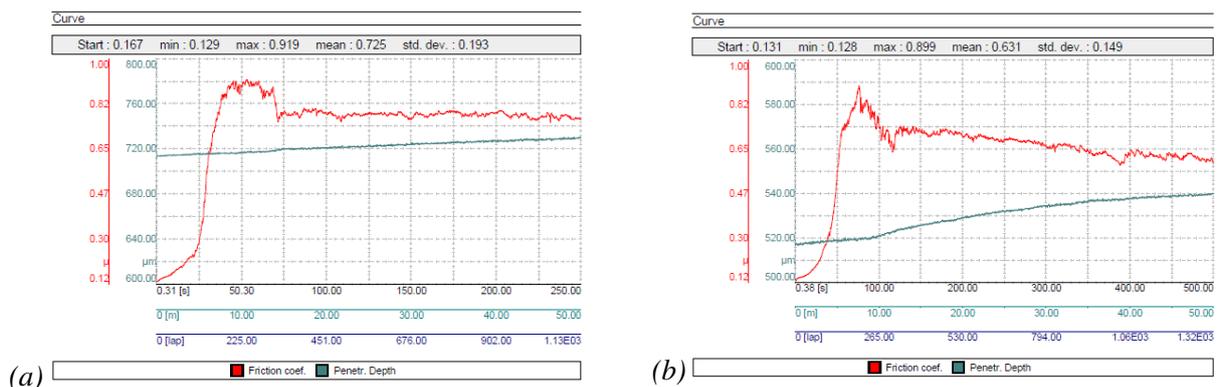


Figure 9. The friction coefficient and penetration depths for AlN coated inconel load normal 1N and linear speed 0,10 m/s (a) and penetration depths for load normal 1N and linear speed 0,20 m/s (b).

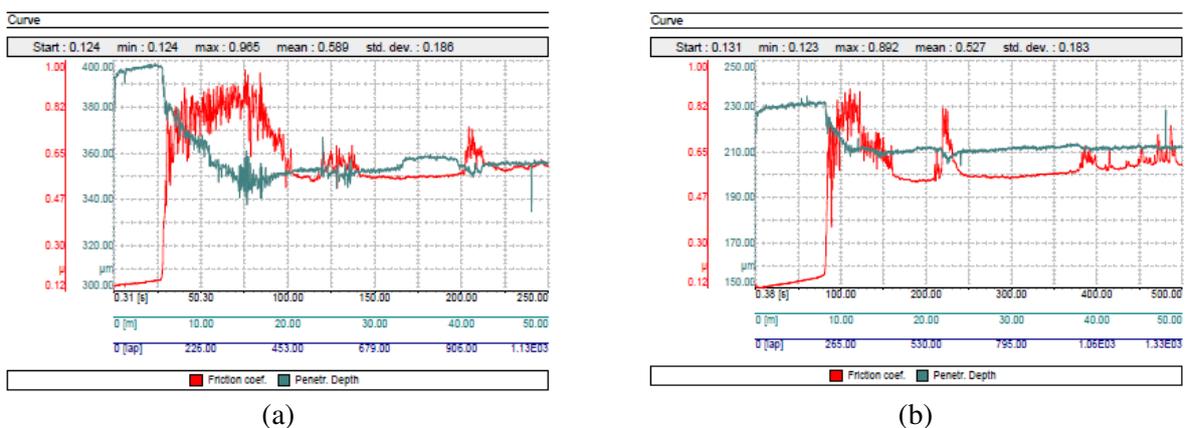


Figure 10. Penetration depths for uncoated Inconel Load normal 1N and linear speed 0,10 m/s (a) and Penetration depths for Load normal 1N and linear speed 0,20 m/s (b).

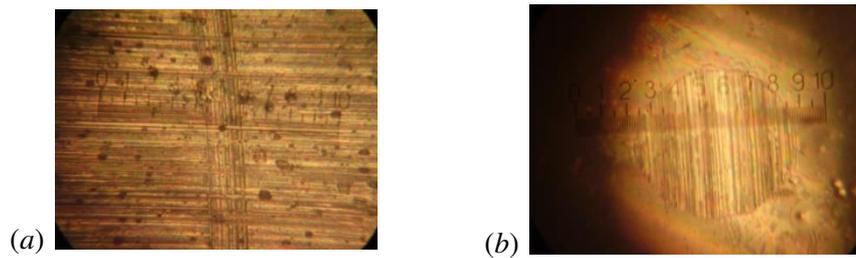


Figure 11. Images of wearing band on disk and wearing on ball, width of wearing band on disk (a); Wearing on ball (b).

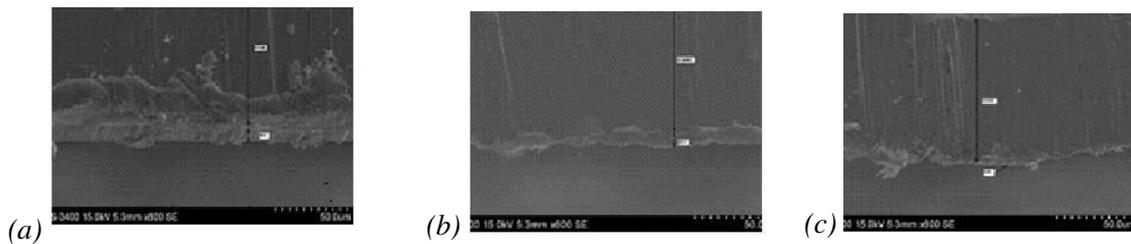


Figure 12. The results tested on CSM Instruments tribometer, initial (a); after 50 seconds (b); after 100 seconds (c).

4. Conclusions

The results of the conducted experiments on the manufactured samples are demonstrating that:

- Usable AlN layers of 1 to 2 microns could be successfully deposited by sputtering directly on the equipment parts needed to be monitored (ball bearings, bearings, rotating axes, cutting tools etc).
- There is recommended to avoid exposing stainless steel components coated with AlN layers at temperatures above 200C⁰. If the temperature is expected to increase over 200C⁰ there is recommended to use an interlayer material or to use a Inconel substrate.
- AlN layers do present a constant high friction coefficient and it isn't recommended to be used on contact surfaces.
- AlN layers cannot withstand harsh abrasion condition therefore it is recommended to avoid using them on contact surfaces.
- There are real possibilities of manufacturing sensors embedded on the monitored components of the machine tool; however, more work has to be done in this direction.

References

- [1] Da Xu L, He W, and Li S 2014 Internet of Things in Industries: A Survey, *IEEE Intl. Conf. Ind. I.* **10** (4) 2233-41
- [2] Parks D A and Tittmann B R 2011 Piezoelectric Material for Use in Harsh Environments *Review of Progress in Quantitative Nondestructive Evaluation AIP Conf. Proc.* **1335** pp 1671-75
- [3] Akiyama M, Tabaru T, and Kishi K 2012 Development of a pressure sensor using a piezoelectric material thin film *Synthesilogy* English edition **5** (3) 162-170
- [4] Fraga M A, Furlan H, Pessoa R S, and Massi M 2014 Wide bandgap semiconductor thin films for piezoelectric and piezoresistive MEMS sensors applied at high temperatures: an overview *Microsyst. Technol.* **20** (1) 9-21
- [5] Stan G E, Pasuk I, Trinca L M, Galca A C, Enculescu M, and Miculescu F 2012 Tilt c axis crystallite growth of aluminium nitride films by reactive rf-magnetron sputtering *Dig. J. Nanomater. Bios.* **7** (1) 41- 50
- [6] Ionescu G et al. 2013 Wireless AlN sensor for condition based monitoring of industrial equipment. In *CAS 2013 vol. 1 (International Semiconductor Conference)* pp. 55-58

- [7] Dumitru V, Morosanu C, Sandu V, and Stoica A 2000 Optical and structural differences between RF and DC Al x N y magnetron sputtered films. *Thin Solid Films* **359** (1) 17-20
- [8] IOFFE Database 2014 AlN – Aluminium nitride. Thermal properties available at: www.ioffe.ru/SVA/NSM/Semicond/AlN/thermal.html
- [9] Agilent Technologies 2012 *Laser and Optics User's Manual* (Ch. 17. Material Expansion Coefficients) available at: psec.uchicago.edu/thermal_coefficients/cte_metals_05517-90143.pdf