# Suppression of Iron Silicide Formation in the Reaction Layer of Sialon – 7.5% Cr Ferritic Steel Joint

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**Abstract.** Diffusion bonding of sialon and 7.5%-Cr Ferritic Steel (FS) in as-received and nitrided condition was studied. Decomposition of sialon and interdiffusion of elements between the sialon and the steel was observed. The interdiffusion of elements produced reaction layers at the joint. The steel moved into the decomposition part of the sialon and replaced the decomposed sialon in this part. Diffusion of silicon into the steel resulted in the silicon-diffusion layer. FeSi<sub>2</sub> was formed in the reaction layer when Si in the decomposition part of the sialon reacted with Fe. The use of nitrided steel in the diffusion bonding had suppressed the formation of the FeSi<sub>2</sub> in the layer.

## Introduction

Reaction layers are usually formed in the joint of ceramic-steel produced by diffusion bonding. The interdiffusion of elements resulted in reaction products which may or may not harm the joint. Silicides are one of the compounds that may be formed in the reaction layer of silicon-based ceramic – metal joint. The compounds are brittle and have different crystal structure with Fe-metal [1]. Therefore, the formation of the compound should be prevented.

The silicide was not formed in the reaction layer of Si<sub>3</sub>N<sub>4</sub> joined with Fe or Fe alloy [1, 2]. It was not found either in the reaction layer of sialon – steel [3]. However, silicides were identified in the joining of Si<sub>3</sub>N<sub>4</sub> with austenitic stainless steel [4, 5]. Formerly, brittle reaction layers were noticed in the joining of the similar materials [6]. Metal silicide also decreased the strength of ceramic and Ti joint [7, 8]. Hence, suppressing the silicide formation could improve the joint. Suppression of the FeSi<sub>2</sub> formation was demonstrated in the joining of Ti and Si<sub>3</sub>N<sub>4</sub> by using nitrogen pre-solved treatment on the Ti prior to joining [7].

The present work investigates the diffusion bonding of sialon (Si-Al-O-N), an engineering ceramic, with 7.5%-Cr Ferritic Steel (FS). Former work recognized the reaction layers at the joint of the sialon with the steel in as received condition [9]. Yet, phase identification on the joint had not been accomplished. In this study, analysis on the phases of the reaction layers of the joint was carried out. Diffusion bonding of the sialon was also conducted with the steel in nitrided condition. This was to study the influence of nitrogen in the steel prior to the diffusion bonding. Formation of the reaction layers and the reaction product was scrutinized. Solution nitriding was applied on the steel to diffuse the nitrogen prior to the diffusion bonding. The joint was characterized using field emission scanning electron microscopy (FESEM), energy dispersive X-ray (EDX) attached to the FESEM, X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS).

## **Experimental Procedure**

The sialon material was  $\alpha$ -sialon provided by Syalon Int. Ltd., UK in the form of discs of 19 mm diameter and 4 mm thickness. The steel was 7.5%-Cr FS in the form of 16 mm diameter rod. Tables 1 and 2 present the chemical compositions of the sialon and the steel, respectively.

Diffusion bonding was carried out using Korea Vac hot press machine of Advance Materials Research Centre (AMREC), Sirim Bhd., Malaysia. The steels to be joined were in as received and

nitrided for four hours conditions. Nitriding for the steel prior to the diffusion bonding has been reported elsewhere [10]. The steel was cut into 1.5 mm thickness and polished with 1  $\mu$ m diamond paste. They were then washed with acetone in an ultrasonic cleaner and dried with a hot air dryer. Joining was performed in sandwich formed where a steel sample was placed in between the two sialon discs. The samples were introduced into the furnace and embedded in boron nitride powder to avoid contact between the steels and the graphite dies set. Uniaxial pressure of 20 MPa was applied while the samples were heated to 1200°C and hold for one hour under vacuum condition of 2 x10<sup>-5</sup> Torr. Heating and cooling rate of the furnace were set at 5°C/minute to avoid thermal shock. The pressure was released when cooling was started.

Table 1. Chemical compositions of sialon [weight percent]

Al	О	N	Si
3.57	2.87	1.58	Balance

Table 2. Chemical composition of as supplied 7.5% Cr FS [weight percent]

С	Cr	Mo	Ni	S	Si	Mn	V	Fe
0.06	7.51	0.69	0.87	0.002	0.32	0.64	0.29	Balance

The diffusion bonded samples were cross sectioned using precision diamond cutter. The cross section surface was ground with diamond grinding discs continued with 600 grade emery papers and polished with 6  $\mu$ m, 3  $\mu$ m and 1  $\mu$ m polycrystalline diamond paste. Vilella etching reagent was used for the sample preparation. Microstructure of the joint was observed using FESEM where EDX analysis was also carried out simultaneously. XRD and XPS examinations were applied on the surface of the reaction layer. For this purpose the diffusion bonded sample was broken to expose the layer surfaces. The examination was then conducted on the surface of the steel from the fractured sample.

## **Results and Discussions**

Diffusion bonding of the sialon with the steels was successfully achieved. Good and sound joint was attained and is shown in the scanning electron micrograph of the joint cross-section (Fig. 1). Two reaction layers were formed at the interfaces. The first was an irregular thin layer adjacent to the sialon, while the second one was thicker and extended into the steel. For the rest of the discussion, terminology of interface layer was used for the first layer, while the later will be called as diffusion layer.

Light precipitates were found throughout the interface layer. This is expected as the reaction product of the sialon with the steel. Whereas, the morphology of the second layer indicated that it was a single phase. In the parent steel, ferritic structure of the as-received samples changed to martensite during the diffusion bonding. Nitrogen diffusion had driven the martensite transformation as it was established in the previous work [10, 11]. Therefore the martensite in this case showed an evidence of the nitrogen diffusion into the area. However, in the sialon – nitrided steel joint, the martensite was also formed in the initial nitrided structure of the steel.

Fig.2a and 2b depict the interdiffusion of elements from the sialon to the steel and vice versa. Sialon decomposed in the interface layer. The decrease of the sialon's elements in this area implied this phenomenon. Silicon was released from the sialon and diffused into the steel. In the sialon – as received steel joint, the silicon reached approximately 60 micron into the steel. It produced the diffusion layer that comprised Fe, Cr and Si. In the joint of the sialon with the nitrided steel, the silicon diffusion distance in the steel was approximately 40 micron. This was shorter than the silicon diffusion distance in the former diffusion bonding. This might suggest that the nitrided steel had reduced the silicon diffusion into the steel.

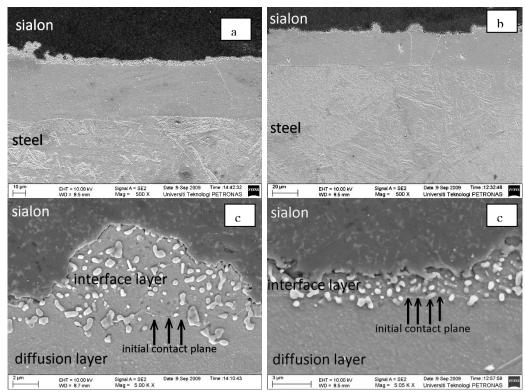


Fig. 1. (a) Cross section of the overall sialon-as received FS joint, (b) Cross section of the overall sialon-nitrided FS joint, (c) the interface layer of sialon diffusion bonded with as-received and (d) the interface layer of sialon diffusion bonded with nitrided 7.5%-Cr FS.

Fe and Cr in the interface layer indicated the diffusion of steel's elements into the sialon and this is shown in Fig. 2. Previous works [2, 3, 5] have recognized that the initial contact of the materials in the diffusion bonding was at the border between the two reaction layers (refer to Fig. 1). This revealed that the interface layer is in the sialon's part. Therefore, the presence of the steel's elements in this part is the evidence of the element diffusion into the sialon. Domination of Fe in this area depicted more-intense diffusion of the steel's elements into the sialon. However, the diffusion of the elements was limited to the decomposed part of the sialon. This is indicated by the absence of the steel's elements in the original sialon.

Analysis of elements was carried out on the several precipitates in the interface layer. Higher concentration of Si in the interface layer of the sialon joined with the as-received steel could be highlighted (Table 3a). In this joint, the precipitates contained maximum of 29.71 weight percent. This is much higher than the Si in the precipitates of the joint of sialon with the nitrided steel (i.e. maximum of 5.73 weight percent).

With the high content of Fe and Si in the precipitates, formation of iron silicides might be expected. This was revealed by the XRD examination attempted on the interface layer surface. The XRD pattern from the interface layer of the sialon diffusion bonded with the as-received steel shows the presence of FeSi<sub>2</sub> (Fig.3). Ferrite ( $\alpha$ ) phase that appeared from the matrix of the layer was also observed. Other than Fe, the matrix also contains Cr and Si that are both ferrite formers [2]; therefore, the phase in the layer matrix is most probably ferrite solid solution with dissolved Cr and Si.

XRD only detected the ferrite phase in the interface layer of the sialon – nitrided steel joint. This was from the matrix layer. Evidently the iron silicide was not formed. Concentration of Si in the precipitates was relatively low. Hence, the XRD result agreed with the elemental analysis of the precipitates in the joint.

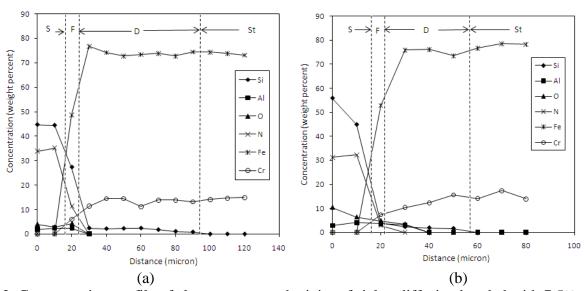


Fig. 2. Concentration profile of elements across the joint of sialon diffusion bonded with 7.5%-Cr FS in (a) as received and (b) nitrided for four hours; S=sialon, F=interface layer, D=diffusion layer; St=steel

Table 3. Concentration of elements of the precipitates [weight percent] in the interface layer of sialon diffusion bonded with (A) as-received and (B) nitrided 7.5%-Cr FS

No	Cr	Fe	Si	О	Al	Y	N
1	0.72	48.47	29.71	2.23	4.91	0	13.96
2	8.56	48.44	26.64	2.30	3.30	0	10.76
3	5.42	49.04	27.37	2.33	4.61	0	11.23
4	7.43	48.37	26.35	2.81	4.31	0	10.72
5	7.75	48.79	26.61	2.41	4.20	0	10.25

No	Cr	Fe	Si	О	Al	Y	N
1	7.51	35.54	5.73	22.05	9.65	19.53	0
2	4.44	65.98	4.38	10.33	5.75	9.11	0
3	15.74	80.08	4.18	0	0	0	0
4	19.32	76.86	2.78	0	1.05	0	0
5	18.69	79.06	2.25	0	0	0	0

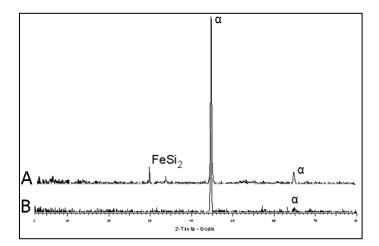


Fig.3. XRD pattern from the surface of the interface layer in the sialon diffusion bonded with (A) as-received and (B) nitrided 7.5%-Cr FS

FeSi<sub>2</sub> in the interface of sialon – as-received steel joint was also revealed by the XPS spectra from the layer (Fig.4a and 4b). This was represented by the peak at 706.92 eV and 719.96 eV that correspond to FeSi<sub>2</sub> [12-15]. Based on the same references, two other peaks at 711.08 eV and 724.58 eV were attributed to Fe<sub>2p3/2</sub> from Fe<sub>2</sub>O<sub>3</sub>. The presence of plasmon loss at around 730 eV in the Fe<sub>2p</sub> spectra indicates the peak of FeSi<sub>2</sub> [13, 14].

The Fe spectra given for the sialon – nitrided steel was very similar (Fig. 4c). Fe in the form of  $Fe_2O_3$  is indicated by the peaks at 711.29 eV and 724.48 eV, respectively. Two peaks at 706.81 eV and 719.8 eV could attribute to  $Fe_{2p3/2}$  peak in the metallic Fe or  $FeSi_2$ . However, plasmon loss was not indicated in the spectra. Therefore those peaks did not correspond to  $FeSi_2$ . It rather represents the Fe in metallic form, since the peak of the chemical states is not much different with the the peak of Fe in  $FeSi_2$  formed, i.e. less than 0.5 eV [13].

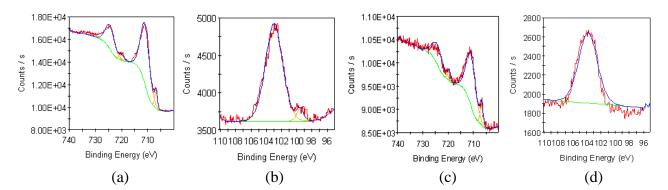


Fig.4. XPS spectra of (a) Fe and (b) Si in the interface layer of sialon – as-received steel and (c) Fe and (d) Si in the interface layer of sialon – nitrided 7.5%-Cr FS

In XPS spectra of Si, FeSi<sub>2</sub> is indicated by the peak at 99.58 eV (Fig. 4b), while the peak at 102.98 eV is Si<sub>2p</sub> from SiO<sub>2</sub> [13-15]. In the other joints, only one peak was shown (Fig. 4d). This is at 103.83 eV which is closed to the peak of Si<sub>2p</sub> from SiO<sub>2</sub>. Therefore, the XPS results confirmed that the FeSi<sub>2</sub> was formed in the joint of the sialon – steel. This also revealed that the iron silicide did not present in the interface layer of the sialon – nitrided steel joint. These are in good agreement with the XRD and the EDX analysis.

The above characterizations proved that the reaction of sialon and as-received 7.5%-Cr FS produced FeSi<sub>2</sub> in the joint interface. They also disclosed that the silicides formation was suppressed in the joint of the sialon with the nitrided steel. Since FeSi<sub>2</sub> is brittle, the nitriding pretreatment on the steel could potentially improve the sialon – steel joint by suppressing the formation of iron silicide.

In summary, sialon decomposed and released Si and N during the interaction with the steel at the diffusion bonding temperature. Both elements diffused into the steel. Silicon diffused into the steel and produced a diffusion layer. Nitrogen diffusion also occurred across the reaction layers. It stimulated the formation of martensite in the parent steel. In the diffusion bonding of the sialon with the as-received steel, more silicon at the sialon-decomposition part reacted with Fe from the steel and precipitated as FeSi<sub>2</sub>. However, nitrided steel used in the diffusion bonding has suppressed the formation of the silicides in the layer. It seems that in this condition more Fe was transferred from the steel into the sialon leading to less concentration of Si in the layer. When reacts with Fe, the composition of Si in the layer might be insufficient to form the silicides. Consequently the formation of the iron silicides was hindered.

## Conclusion

During the diffusion bonding with 7.5%-Cr FS, sialon decomposed and liberated silicon and nitrogen that subsequently diffused into the steel. Diffusion layer in the steel was resulted due to the silicon diffusion. Nitrogen also diffused into the steel and formed martensite. The steel's element diffused in significant quantity into the sialon and filled up the sialon-decomposition area to produce the interface layer. FeSi<sub>2</sub> precipitated in the interface layer. This is the reaction product of the sialon and the as-received steel during the diffusion bonding process. The used of the nitrided steel has been proven to suppress the formation of FeSi<sub>2</sub> in the interface layer of the joint with sialon. Thus, the brittle fracture at the joint can be avoided.

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### **Preface**

This special issue of Defect and Diffusion Forum contains selected refereed papers presented at the Sixth International Conference on Diffusion in Solids and Liquids (DSL-2010) held at the Hotel Concorde La Fayette Paris, France during the period 5<sup>th</sup>-7<sup>th</sup> July, 2010.

The goal of the conference was to provide a unique opportunity to exchange information, to present the latest results as well as to review the relevant issues on contemporary diffusion research. Young scientists were especially encouraged to attend the conference and to establish international networks with well-known scientists.

Several topics were highlighted in the scope of special sessions and some of the presented papers are collected in this volume: Characterization and Properties of Hard Coatings (by I.E. Campos Silva, R.E. Galindo), Diffusion, Permeability and Gas Dissolution in Polymers and Polymer Foams (E. Solórzano Quijano, M. Angel Rodríguez Pérez), Grain Boundaries and Interfaces: Structure, Thermodynamics and Diffusion Properties (V.V. Popov, B. Straumal), Active Biomaterials and Tissue Engineering (N. Jessel, A. Bianco, V. Ball), Carbon and Oxide Based Nanostructured Materials (N. Yahya, M. Rusop Mahmod, K. Koziol), Diffusion in Solid Ionic Conductors (P. Heitjans, G.E. Murch, I. Belova), Heat and Mass Transfer in Porous Media (J.M.P.Q. Delgado), Microstructural Development of Metallic Materials Through Diffusion Controlled Processes (Y. Houbaert, L. Suárez, P. Rodríguez), Hydrogen-Related Kinetics in Materials (Y. Fukai, M. Wilde) and Computational Heat and Mass Transfer: New Approaches and Innovative Schemes (R. Bennacer, M. EL Ganaoui, L. Tadrist).

More than 340 scientists and researchers coming from more than 56 countries attended the conference. The large number of presented papers emphasises the considerable academic and industrial interest in the conference theme. The editors wish to thank the authors and delegates for their participation and cooperation, which made this sixth conference especially successful.

Finally, we wish to express our warm thanks and appreciation to our colleagues and associates for their sustained assistance, help and enthusiasm during the preparation of the conference.

The seventh conference, DSL-2011, will be held in Algarve, Portugal, from 27-29 June, 2011 (http://www.dsl2011-algarve.com/).

March 2011

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## **Table of Contents**

**Preface and Organizing Committees** 

Effect of Catalyst Diffusion Coefficient on Ethylbenzene Dehydrogenation M.E. Zeynali and F. Mohammadi	1
Investigation of the Effect of Diffusion Process in the Catalyst Pellet on Overall Reaction Rate of Dehydrogenation of Diethylbenzne to Divinylbenzne M.E. Zeynali	7
Diffusion Layers with Ti and Ti+Al Formed on 316L Austenitic Steel by a Pack Cementation Procedure	•
I.M. Britchi, M. Olteanu, N. Ene and P. Nita	13
Influence of Important Nanoemulsions pH on Performance of Nanostructures Catalysts for H <sub>2</sub> Production in Syngas Reactions Z. Fakhroueian, P. Esmaeilzadeh, N. Afroukhteh Langroudi, H. Varmazyar, M. Ahmadirad, P.E.	
Zadeh, M. Yousefi, M. Karami, A. Shafiekhani and S. Sepehriseresht  Cosmic Censorship!: Thermal Transport in a 'Naked' Black Hole  D. L. Complete and T. L. Angelon Branch and T. A. Marie and T. Marie and T	20
R.L. Corral Bustamante, A.R. Rodríguez-Corral, T.J. Amador-Parra and E.A. Vázquez-Tapia  Assessment of Thermal Non-Equilibrium Condition on Heat Transfer through a Channel  Lined with Porous Media – Constant Wall Temperature	27
M. Abkar, P. Forooghi and A. Abbassi	33
Computational Algorithms for Topological Cycle Indices of Tert-Butyl Alcohol by Computational Science N. Ghafooriadl, S. Asadzadeh Olghi and A. Moghani	39
Comparison of the Breakthrough Curves Obtained by ASTM D2007-03 Annex 1 and on a Bench Unit for the Selection of Adsorbents for the Removal of Nitrogen and Sulfur Contaminants from Fuels	
M.A.G. Figueiredo, G.V. Estrela, W.C. Souza and S.S.X. Chiaro	45
Application of Thermo-Mechanical Process to Achieve Nanostructure in 301 Austenitic Stainless Steels A. Shokohfar, S.M. Abbasi, A. Yazdani and B. Rabiee	51
Phase Transformations on ASTM a 744 Gr. CN3MN Superaustenitic Stainless Steel after	
Heat Treatment M. Ritoni, M. Martins, F.C. Nascimento and P.R. Mei	56
Calculation of the Diffusion Coefficient of Uranium in Compacted Clay: Effect of the Temperature S. Korichi, M. Keddam and A. Bensmaili	64
Surface Characterization of a Nitrided Low Alloy Steel	04
M. Keddam, R. Kouba, R. Chegroune and B. Bouarour	70
Formation of Zirconia and Titania Nanotubes in Fluorine Contained Glycerol Electrochemical Bath	7.0
Z. Lockman, S. Ismail, G. Kawamura and A. Matsuda	76
Grain Boundary Parameters in Sandstone and Limestone V. Palchik	82
Electrochemical Mass Transfer Measurements of CO <sub>2</sub> in MDEA Solutions R. Maceiras, X.R. Nóvoa, A. Cancela and E. Álvarez	87
Simulation of VOCs Recovery Process by Absorption R. Maceiras, S. Urrejola, A. Cancela, A. Sanchez and J. Díaz	93
The Effect of Stabiliser's Molarity to the Growth of ZnO Nanorods Z. Khusaimi, M.H. Mamat, M.Z. Sahdan, N. Abdullah and M. Rusop	99
Aligned Growth of Zinc Oxide Nanorods on Catalyst-Seeded Si Substrate by Aqueous-Solution Immersion Method Z. Khusaimi, M.H. Mamat, M.Z. Sahdan, N. Abdullah and M. Rusop	104
Microstructure-Toughness Relationship in AISI4340 Steel	201
N. Saeidi and A. Ekrami	110
Studies of Ionic Conductivity and Dielectric Behavior in Polyacrylonitrile Based Solid Polymer Electrolytes  Z. Osman, K.B.M. Isa, L. Othman and N. Kamarulzaman	116
2. Commi, 11.2.11. 100, 2. Cumum und 11. 12mmunul2mmun	110

Hydrodynamic Simulation of Drift Mobility in N-Hg <sub>0.8</sub> Cd <sub>0.2</sub> Te M. Daoudi, A. Belghachi, L. Varani and C. Palermo	122
Composition Dependent Diffusivities in Multi-Component Systems B. Wierzba and M. Danielewski	127
In Situ Simulation by RHEED and Photoemission of GaAs (001) β <sub>2</sub> (2x4) Reconstructed	
Surface H. Khachab, Y. Abdelkafi and A. Belghachi	132
<b>Determination of PBP by Using a Nano SiO2/GC Modified Electrode</b> A. Shokuhi Rad	138
Molecular Dynamics Simulation of Solidification of Ag-x%Au Nanoalloy J. Davoodi and L. Mehri	143
<b>Diffusion of Hydrogen in Amorphous Ni-Zr Alloys</b> E.A. Pastukhov, N.I. Sidorov, A.A. Vostrjakov and V.P. Chentsov	149
Effect of the Latent Heat on Wax Deposit in Pipelines R. Boucetta and M. Kessal	154
<b>Polyol-Mediated Synthesis of TiO</b> <sub>2</sub> <b>Nanoparticles</b> J.W. Kang, D.H. Kim, V. Mathew, J.H. Gim, I. Yu, C.H. Woo, E.J. Kim, C.H. Choi and J.K. Kim	160
Study of Semisolid and ECAP Processes on Al-Fe-Si Alloy - Microstructure and Kinetic Grain Growth	
D. Azimi-Yancheshmeh and M. Aghaie-Khafri	166
Phase and Amplitude Control of Optical Properties of Quantum Dot Molecules M. Mahmoudi and S. Goharshenasan	172
Heat Transfer in Nanocomposites with Monte-Carlo Simulations K. Bui and D.V. Papavassiliou	177
The Influence of Rubber Blend Aging and Sample Homogeneity on Heat Transport	
<b>Phenomena</b> P. Koštial, Z. Jančíková, I. Ružiak, I. Kopal and P. Jonšta	183
Experimental and Theoretical Research of the Shell Side Heat Transfer Coefficient and Pressure Drop in a Plastic Shell and Tube Heat Exchanger R. Aguilar Osorio and K. Cliffle	187
Raman Spectroscopy: Mutual Diffusion Coefficient in Hydrogels A.A. Naddaf and H.J. Bart	193
Characterization of Nickel Based Alloys (Hard Facing Alloys) with Additions of Ti and Mo K. Khenfer, S. Lebaili and S. Hamar-Thibault	199
Effectiveness Parameters for the Heat and Mass Transfer in a Desiccant Wheel C.R. Ruivo, J.J. Costa and A.R. Figueiredo	205
Thin Scandium Layer on Solid Aluminium: Thermodynamic Investigation A.B. Shubin, K.Y. Shunyaev and L.F. Yamshchikov	211
<b>Dynamic Thermal Diffusivity Measurements – A Tool for Studying Gas-Solid Reactions</b> H.M. Ahmed, N.N. Viswanathan and S. Seetharaman	217
Phase Transformation and Thermoelectric Properties of Sn-Filled/Fe-Doped CoSb <sub>3</sub>	
Skutterudites I.H. Kim	223
A Study on the Coupled Thermal and Damage Effects in Deforming Solids M. Vaz, P.A. Muñoz-Rojas and M.R. Lange	229
<b>Pulse Electrolysis for the Production of Hard Ni/ZrO</b> <sub>2</sub> <b>Composite Coatings</b> M.M. Dardavila and C. Kollia	235
Finite Element Modeling of Silicon Transport into Germanium Using a Simplified Crystal Growth Technique F. Mechighel, M. El Ganaoui, B. Pateyron, M. Kadja and S. Dost	240
Effects of Mechanical Activation during the Synthesis of Tungsten Carbide Powders by Carbothermic Reduction of Tungsten Oxide J. Ma, S.G. Zhu, H. Ding and W.S. Gu	248
Numerical Analysis of Solute Segregation in Directional Solidification under Static Magnetic Field S. Nouri, M. Benzeghiba and A. Benzaoui	253
•	

Effect of Quincke Rotation and Taylor Circulation on Mass Transfer from a Fluid Drop in a Constant Electric Field	
T. Elperin and A. Fominykh	259
Quantitative Evaluation of the Raman Spectra of Carbon Layers M. Golabczak and A. Konstantynowicz	265
Grain Structure Evolution of Mg Alloy AM60 Influenced by Ca Addition H. Hu, Q. Zhang and X.P. Niu	271
Solidification of Discontinuous Al <sub>2</sub> O <sub>3</sub> Fiber Reinforced Magnesium (AM60) Matrix Composite	
Q. Zhang, H. Hu and J. Lo	277
The Influence of Acetic Acid on High Temperature Corrosion of Selected Steels Utilized in Automobile Industry  Z. Grzesik, M. Migdalska and S. Mrowec	283
Effect of Multifilamentary Nb/Cu-Sn Wire Diameter on the Nb <sub>3</sub> Sn Diffusion Layers Structure	203
E.N. Popova, E.P. Romanov, I.L. Deryagina, S.V. Sudareva, E.A. Dergunova, A.E. Vorobyova and S.M. Balaev	289
Origin of "Inverse Isotope Effect" of Hydrogen Diffusion in Palladium O. Yoshinari	295
Physical Properties of Diffusive-Hardening Alloys of Copper with Gallium and Tin A.B. Shubin, K.Y. Shunyaev, V.A. Bykov and S.I. Noritsin	301
The Effect of the Microstructure on Oxidation of Sulfide-Metal Alloys of Copper and Nickel E.N. Selivanov, R.I. Gulyaeva, L.Y. Udoeva and A.D. Verchinin	306
Synthesis of W- Cu- Ag Nanopowders Produced by a Co-Precipitation Process G. Taghavi, H.R. Rezaie and H. Razavizadeh	312
Production of Cu/SiC Nanocomposite Layers by Friction Stir Processing M. Barmouz, M.K.B. Givi and P. Asadi	319
Comparison of Corrosion Performance of Zn and Zn/Al 85/15 Coatings Produced by TWEA	
A. Güleç, F. Yilmaz, A. Türk and F. Üstel	325
Thermo-Mechanical Behaviour of Ultrahigh Molecular Weight Polyethylene-Carbon Nanotubes Composites under Different Cooling Techniques S. Kanagaraj, A. Fonseca, R.M. Guedes, M.S.A. Oliveira and J.A.O. Simões	331
Carbon Fiber-Reinforced Composites in Automotive Engine R.E. Farsani and A. Shokuhfar	341
The Effect of the Pyrolysis Furnace Type on the Yield of Silicon Carbide Whiskers Produced from Rice Husks	
R.E. Farsani, F. Akhlaghi and A. Sedghi	346
<b>Experimental Study of Enhanced Pool Boiling Heat Transfer Using Graphite Foam Inserts</b> K.C. Leong, L.W. Jin, I. Pranoto, H.Y. Li and J.C. Chai	352
Acoustical Parameters: Aqueous Solution of Borassus Flabellifier Fruit Pulp by Non Destructive Method B. Shrivastava, A. Mishra and S. Phadke	358
Estimation of Effective Moisture Diffusivity in Starchy Materials Following Hydrothermal	336
Treatments S.A. Bahrani, C. Loisel, J.Y. Monteau, S.A. Rezzoug and Z. Maache-Rezzoug	364
Synthesis of Cobalt Nano Particles on Silica Support Using the Strong Electrostatic Adsorption (SEA) Method	270
K.L. Chee, N.A. Mohd Zabidi and M.S. Chandra  Correction Polymer of Legen Nitrided Ti (ALAY Allege A Portion)	370
Corrosion Behavior of Laser Nitrided Ti-6Al-4V Alloy: A Review R.S. Razavi, G.R. Gordani and H.C. Man  Mathematical Modeling of Heat Transfer in Laser Surface Headering of AlSI 1050 Steel	376
Mathematical Modeling of Heat Transfer in Laser Surface Hardening of AISI 1050 Steel R.S. Razavi, G.R. Gordani and S. Tabatabaee	381
Reactive Diffusion at the Cylindrical Dissolving of Copper in the Solder Melt J. Drápala, A. Struhařová, D. Petlák, V. Vodárek and P. Kubíček  Electron Diffusion in 700 Nonconstantials And A. Lunadana Lunatiantian	387
Electron Diffusion in ZnO Nanomaterial: An Ac Impedance Investigation R. Rusdi, N. Kamarulzaman, M. Nor Sabirin, Z. Osman and A.A. Rahman Missesters and Changes of FSV 414 Supersular decision TLP Bondies	393
Microstructural Changes of FSX-414 Superalloy during TLP Bonding R. Bakhtiari and A. Ekrami	399

Macrosegregation and Porosity during Directional Solidification of a Ternary Al-9wt%Si-3wt%Cu Alloy	
L.G. Gomes, P.D. Jácome, I.L. Ferreira, D.J. Moutinho, O.L. Rocha and A. Garcia	405
Diffusion Paths in the Ternarysystem Fe-Co-Ni: An Empirical Approach J. Priimets and Ü. Ugaste	411
Self-Diffusion Coefficient of Molecular Fluid in Porous Media V. Andryuschenko and V. Rudyak	417
Sonochemical Synthesis and Characterization of Hydroxyapatite Nanoparticles M. Mohebali and A. Shokuhfar	423
Numerical Comparison of Two and Three Dimensional Flow Regimes in Porous Media A.A. Abbasian Arani and M. Dehghani	427
Free Convection in a Nanofluid Filled Square Cavity with an Horizontal Heated Plate A.A. Abbasian Arani, M. Mahmoodi and M. Amini	433
<b>Microstructure Characterization of TBC Layers of Gd<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub> Type</b> G. Moskal	439
Thermal Diffusivity of TBC Layers of RE <sub>2</sub> Zr <sub>2</sub> O <sub>7</sub> Type G. Moskal	445
High Temperature $SO_2\text{-}Gas$ Corrosion of Ni-Cr-Co Base Superalloy between 800 and $1000^{\circ}\mathrm{C}$	
S.H. Bak and D.B. Lee	451
Modeling of Transient Moisture Diffusion in Wood below the Fiber Saturation Point J. Eitelberger and K. Hofstetter	455
Fabrication of Multi-Walled Carbon Nanotube/Vinyl Ester Nanocomposites: Dispersion and Stabilization	
M. Chitsazzadeh, H. Shahverdi and M.M. Shokrieh	460
Diffusion in Co-Ni System Studied by Multifoil Technique V.D. Divya, U. Ramamurty and A. Paul	466
Microstructure and Hydrogenation Property of Hyper-Eutectic Mg-Ni Alloys H. Saitoh, M. Kondo and H. Nakamura	472
Numerical Investigation of Impingement Heat Transfer Enhancement on a Flat Plate with an Attached Porous Medium P.S. Wu, Y.W. Lo and F.C. Cheng	477
Oxidation Behaviour of WE54 and Elektron 21 Magnesium Alloys	
A. Kiełbus, T. Rzychoń and R. Przeliorz	483
The Influence of Heat Treatment Parameters on the Thermal Diffusivity of WE54 and Elektron 21 Magnesium Alloys A. Kiełbus, T. Rzychoń and G. Moskal	489
Analytical Solution for Solute Transport in Semi-Infinite Heterogeneous Porous Media B.Q. Deng, Y.F. Qiu and C.N. Kim	495
Effect of SiC Volume Fraction on the Microstructural and Mechanical Properties of Cu/SiC Composite Layer Fabricated by FSP	.,,
P. Asadi, M.K.B. Givi and M. Barmouz	500
In Situ Analysis of Hydrogen Mobility during Hydrogen Permeation through Nb-Based Hydrogen Permeable Membranes H. Yukawa, T. Nambu and Y. Matsumoto	506
Characterization of Hydrophilic Membranes Based on PVP and Carboxymetylcelulose	300
Staple Fibers Infused with Calcium Alginate L.F. de Miranda, M.C. Terence, I.T. Ferro Barbosa, I. Martins Rubin and J.F. Alves	513
Investigation of a New Sn-Cu-Ga Alloy Solder M. Matsushita, Y. Sasaki and Y. Ikuta	518
Gas Nitriding of Sintered Austenitic Stainless Steel J. Baranowska and V. Amigó	524
Kinetics of Low Temperature Nitriding of Precipitation Hardened Stainless Steel P. Kochmański and J. Baranowska	530
Numerical Study of Magnetic Field Effects on Buoyancy Driven Convection in a Non-	
Isothermally Heated Square Enclosure G.A. Sheikhzadeh, M. Pirmohammadi, A. Fattahi and M.A. Mehrabian	536

Microstructure and Properties of CBN Diffusion Coating on High-Speed Steel A.S. Chaus, L. Čaplovič and J. Porubský	542
Confined Bubble and Heat Transfer during Flow Boiling in a High Aspect Ratio Mini-Channel	
Y. Wang and K. Sefiane	548
Partition of Free Fatty Acids in Deacidification of Macaúba Pulp Oil by Liquid-Liquid Extraction Using Ethanol/Water as Solvent R.G. Brito Mariano, C.M. Da Silva, S. Couri, R.I. Nogueira and S.P. Freitas	554
Reducing Hydrogen Permeation through Metals	334
M. Dapor and A. Miotello	560
Influence of Measurement Results of Thermal Conductivity and Heat Transfer Coefficients on the Simulation Results of Casting Process of Aircraft Engine Elements G. Moskal, J. Cwajna, B. Witala and R. Cygan	566
Characterization of Microstructure and Properties of Plasma Sprayed Ceramic Coatings on AZ91 Magnesium Alloy	571
G. Moskal, A. Iwaniak and B. Witala	571
Characteristics of Thermal Properties of Gd <sub>2</sub> Zr <sub>2</sub> O <sub>7</sub> – ZrO <sub>2</sub> XY <sub>2</sub> O <sub>3</sub> Powder Mixtures Intended for Deposition of Gradient Layers of TBC Type G. Moskal, L. Swadźba and B. Witala	577
Characteristics of Phenomena in Powders Type RE <sub>2</sub> Zr <sub>2</sub> O <sub>7</sub> -Al <sub>2</sub> O <sub>3</sub> in High Temperature Annealing Conditions	
G. Moskal, L. Swadźba, M. Hetmańczyk and B. Witala	583
The Impact of Diffusion Process on the Interface Microstructure in AZ91 Magnesium Matrix Composites Reinforced with TiN Nanolayer Modified Carbon Fibres  A. Olszówka-Myalska	589
High-Temperature Diffusion Barrier for Ni-Cr Cast Steel	309
A. Kochmańska and M. Garbiak	595
Real Time Prognostic Strategies: Application to Gas Turbines M. Yadegari	601
Investigation of the Effect of Electrospun Polyethersulfone Nanofibers in Membrane S. Shahgaldi, Z. Yaakob, M. Ghasemi, W.R. Wan Daud and D.J. Khadem	607
<b>Synthesis and Characterization of PES/TiO<sub>2</sub> Nanofibers Membrane</b> D.J. Khadem, Z. Yaakob, S. Shahgaldi, M. Ghasemi and W.R. Wan Daud	613
Weight Analysis in a Quantum Mechanical Transport Model for Nano and Bio Materials P. Di Sia and V. Dallacasa	620
A New Approach for the Diffusion Coefficient Evaluation of Sulfur in CaO-SiO <sub>2</sub> -Al <sub>2</sub> O <sub>3</sub> Slag L. Muhmood, N.N. Viswanathan and S. Seetharaman	626
Experimental Results on Evaporation Waves J.C. Graña-Otero and I.E. Parra	635
Modeling of Heat and Mass Transfer in an Annular Adsorber for Solar Cooling Machine: Performance Coefficients	032
N. Allouache, R. Bennacer, S. Chikh, A. Al Mers and N. Mimouni	641
Characteristics of Liquid–Solid Mass Transfer in a Bubble Column Equipped with a Vertical Tube of Circular Fins	
H.S. Bamufleh, S.A. Nosier and M.A. Daous	647
Effect of Ti and Fe Alloying on Kirkendall Voids Feature in Ni/Ni <sub>3</sub> Al Welded Joints after the High Temperature Annealing M. Losertová, J. Drápala and J. Vrbický	653
Influence of the Thermo-Piezo-Viscous Effect on the THD Lubrication in Porous Self- Lubricating Bearings	
S. Boubendir, R. Bennacer and S. Larbi	659
Simplified Convective Heat Transfer in Vertical Airflow Regimes with Shading Device M. Haase and A. Amato	665
Numerical Simulations of Heat Transfer in Plane Channel Flow N. El Gharbi, R. Absi and A. Benzaoui	671
Thermal Properties of Zirconium Oxide-Based Ceramic Targets Modified with Oxides of Rare Earth Elements	
M. Kostecki and A.R. Olszyna	676

Mechanisms of Microstructural Rearrangement on Two-Dimensional Aggregates under Compressive Stress	600
S. Promkotra	682
Microstructural Evolution of Synthesized Polyhydroxyalkanoates (PHAs) Corresponding to their Blends P. Kaewkannetra and S. Promkotra	688
Improving the Diffusion Controlled Cementation of Cadmium Ions Using Reciprocating Fixed Bed of Zinc Rings	
A.H. El Shazly, A.A. Mubarak and H.S. Bamufleh	694
Augmentation of the Performance of Batch Electrocoagulation Unit by Using Gas Sparging A.H. El Shazly	700
Structure and Thermal Transformations of Hydrated Magnesium Silicates E.N. Selivanov, S.V. Lazareva, L.U. Udoeva and R.I. Gulyaeva	708
Heat and Mass Transfer Made Visible M.F.M. Speetjens and A.A. Van Steenhoven	713
The Influence of Phase Dispersion of Sulfide Alloys on their Electrochemical Properties E.N. Selivanov, O.V. Nechvoglod and S.V. Mamyachenkov	719
Influence of Geometry on the Position and the Intensity of Maximum Kinetic Energy in a Combustion Chamber	
A.C. Stuparu and S. Holotescu	725
An Overview of the Diffusion Studies in the V-Si System S. Prasad and A. Paul	731
Nutrients and Metabolic Secretions Transfer in Cornea H. Dória Silva, L.C. Pereira Dória, C. Pereira Dória, C. Pereira Dória, M.d.C.T. Dória Silva, H. Dória Silva Júnior and M.T. Dória Silva	737
<b>Structural, Mechanical and Magnetic Properties of Mechanically Alloyed Fe</b> <sub>40</sub> Co <sub>60</sub> <b>Powders</b> F. Otmane, S. Bergheul, M. Zergoug and M. Azzaz	743
New Use Heat Transfer Theories for the Design of Heat Setting Machines for Precise Post- Treatment of Dyed Fabrics R.W.L. Ip and E.I.C. Wan	748
Diffusion of Particles Adsorbed on a Dynamically Disordered Reconstructive Surface A. Tarasenko and L. Jastrabik	752
Sensitivity Tests of Simulation Models Used in Chosen Calculation Codes on Uncertainty of Thermo-Mechanical Parameters during Virtual Mechanical Stress Estimation for Ferrous Alloy Castings	
Z. Ignaszak and P. Popielarski	758
Estimation of Coupled Thermo-Physical and Thermo-Mechanical Properties of Porous Thermolabile Ceramic Material Using Hot Distortion Plus® Test Z. Ignaszak, P. Popielarski and T. Strek	764
Uncertainty of the Diffusion Measurements on Scaffolds for Cell Growth	
G. Sassi, M. Bernocco and M. Sassi  Effect of Co. Daving of Nano Silver Content and Nitrogen on Titania on the Visible Light	770
Effect of Co-Doping of Nano Silver, Carbon and Nitrogen on Titania on the Visible Light Activity for Methylorange Degradation N.N. Binitha, Z. Yaakob, P.P. Silija, P.V. Suraja and S.M. Tasirin	776
<b>Modeling of Non-Uniform Thermoelement Devices Subject to Lateral Heat Convection</b> R. Kafafy and A. Hameed	782
Microstructure and Properties of Diffusion Boride Layer on Die Steel M. Beznák, A.S. Chaus and L. Čaplovič	788
<b>Turbulent Diffusion of Suspended Particles: Analysis of the Turbulent Schmidt Number</b> R. Absi, S. Marchandon and M. Lavarde	794
Polymorphic and Morphological Transformation during the Transition from a Propagating Band to Static Bands in the Nickel Hydroxide/Ammonia Liesegang System M. Al-Ghoul and M. Ammar	800
Oxidation and Mechanical Behaviour of Iron Based Alloys and Nickel Based Alloy for Spring Like Interconnects of the PRODHYGE High Temperature Electrolyser M. Lamazouade-Tarek, I. Aubert and J. Jumel	806
Determination of the Pore Sizes and their Influence on the Capillary Imbibition into Glass	
Wool  L. Marmoret, H. Beji and A. Perwuelz	812

Sorptivity and Hydraulic Diffusivity of Coating Gypsum and Impact of Hemp Fibers Addition	
S. Gassita, L. Marmoret, H. Ben Hamed and H. Beji	818
The Influence of Rare Earth, Strontium and Calcium on the Thermal Diffusivity of Mg-Al Alloys	
T. Rzychoń and A. Kiełbus	824
Determination of Gamma Transmittance and Density Assessment for Al Doped ZnO Thin Films by Using Gamma Transmission Technique	
B. Karagoz, O. Ozdemir, O. Karacasu, N. Baydogan, H. Çimenoğlu, E.S. Kayali, A.B. Tugrul, N. Altinsoy, S. Haciyakupoğlu, S. Kiziltas, M. Okka, G. Albayrak, H. Sengel, F. Akmaz and A. Parlar	830
Electromagnetic Radiation Effect on ZnO Nanocrystallites L. Sepet, N. Baydogan, H. Çimenoğlu, E.S. Kayali, A.B. Tugrul, N. Altinsoy, G. Albayrak, H. Sengel, F. Akmaz and A. Parlar	836
Single Layer Drying Behavior of Apple Slices in a Microwave Dryer S. Çelen, K. Kahveci and U. Akyol	842
Drying Behavior of Polyester Based Yarn Bobbins in a Hot-Air Bobbin Dryer A. Cihan, K. Kahveci, U. Akyol and D. Akal	848
Simulation of Drying Behavior of Cotton Bobbins by a Simultaneous Heat and Mass Transfer Model	
U. Akyol, K. Kahveci, A. Cihan and D. Akal	854
Finite Element Simulation of Drying of Rough Rice O. Hacıhafızoğlu, A. Cihan and K. Kahveci	860
Airborne Pathogens Transport in an Aircraft Cabin M.B. Acikgoz, B. Akay, A.F. Miguel and M. Aydın	865
Fluid Flow and Solid/Fluid Suspensions Flow in 3-D Packed Beds of Spheres: The Effect of Periodicity of Fixed Beds	
A. Serrenho and A.F. Miguel	871
Micro Arc Oxidation of AZ91 Mg Alloy for Improved Corrosion Resistance H.G. Yavuz, M. Gunyuz, I.M. Ozkara, M. Baydogan and H. Çimenoğlu	877
Electrochemical Hydriding of Mg-Based Alloys D. Vojtěch, V. Knotek and P. Novák	882
Suppression of Iron Silicide Formation in the Reaction Layer of Sialon – 7.5% Cr Ferritic Steel Joint	
F. Hudiyo, P. Hussain and O. Mamat	888
Elaboration of Sol-Gel Derived Hydroxyapatite / Yttria Stabilized Zirconia Composite Coatings Obtained for Biomedical Application S. Salehi and M.H. Fathi	894
Effect of Temperature on the Growth of Vertically Aligned Carbon Nanotubes from Palm Oil	071
S. Abu Bakar, S. Muhamad, P.S. Mohamad Saad, R. Md Nor, Y. Mohd Siran, S.A.M. Rejab, A.J. Asis, S. Tahiruddin, S. Abdullah and M.M. Rusop	900
The Effect of Precursor Vaporization Temperature on the Growth of Vertically Aligned Carbon Nanotubes Using Palm Oil	
S. Abu Bakar, S. Muhamad, P.S. Mohamad Saad, S. Afif Mohd Zobir, R. Md Nor, Y. Mohd Siran, S.A.M. Rejab, A.J. Asis, S. Tahiruddin, S. Abdullah and M.M. Rusop	906
Speckle-Based Deflection Techniques in Diffusivity Measurements D. Ambrosini, D. Paoletti, R. Di Biase and G. Galli	912
Role of Forming Gas Annealing Characteristics on the Selective Surface Oxidation of Fe- Mn-Si-Cr Model Alloy S. Swaminathan and M. Rohwerder	918
Effect of Impinging Jet on Heat Conduction in Workpieces Irradiated by a Moving Heat	
Source N. Bianco, O. Manca, S. Nardini and S. Tamburrino	924
Dispersion of Twin Inclined Fume Jets of a Variable Height within a CrossFlow A. Radhouane, N. Mahjoub Said, H. Mhiri, G. Le Palec and P. Bournot	929
Non-Isothermal Enhanced Recovery of Heavy Oils by Numerical Simulation A. de Lima Cunha, S.R.F. Neto, A.G.B. de Lima and E.S. Barbosa	935

Numerical Investigation of the Thermal Properties of Irregular Foam Structures S.M. Hossein Hosseini, A. Kharaghani, C. Kirsch and A. Öchsner	941
Performance of a Water Ammonia Absorption System Operating at Three Pressure Levels N. Bouaziz, R. Ben Iffa, L. Kairouani, S. Chikh and R. Bennacer	947
3-D Finite Element Simulation of Friction Stir Welding Process of Non Similar Aluminum-	
Copper Sheets A. Alimoradi, M. Loh-Mousavi and R. Salekrostam	953
Numerical Simulation of Transient Temperature Fields in Solids Irradiated by Moving	
Gaussian and Donut Sources O. Manca, S. Nardini, D. Ricci and S. Tamburrino	959
Flow Structure Issued from a Bent Chimney around a Cylindrical Obstacle: Effect of the	
Aspect Ratio I. Bhouri Baouab, N. Mahjoub Said, H. Mhiri, G. Le Palec and P. Bournot	965
Heat and Mass Diffusion Including Shrinkage and Hygrothermal Stress during Drying of	
Holed Ceramics Bricks J. Barbosa da Silva, G.S. Almeida, W.C.P. Barbosa de Lima, G.A. Neves and A.G.B. de Lima	971
Effect of Tool Nose Radius on Nano-Machining Process by Molecular Dynamics Simulation S. Vahid Hosseini, M. Vahdati and A. Shokuhfar	977
Investigation of Interatomic Potential on Chip Formation Mechanism in Nanometric	,,,
Cutting Using MD Simulation	002
S. Vahid Hosseini, M. Vahdati and A. Shokuhfar	983
Oxygen Diffusion in Yttria-Stabilized Zirconia Subjected to Mechanical Stress W. Araki and Y. Arai	989
Low Temperature Nitriding of a Martensitic Stainless Steel R. Karadas, O. Celik and H. Çimenoğlu	994
The Effect of Micro Arc Oxidation Process Parameters on Surface Properties of Ti6Al4v	
Alloy M. Gunyuz, M. Baydogan, H. Çimenoğlu and E.S. Kayali	1000
Friction and Wear Behaviour of Borided Ti6Al4v Alloy Sliding Against Al <sub>2</sub> O <sub>3</sub> and Si <sub>3</sub> N <sub>4</sub>	
Balls in Water E. Atar, E.S. Kayali and H. Çimenoğlu	1004
Nitriding of Cp Titanium	
F. Seahjani and H. Çimenoğlu	1010
Effect of Tube Bending on Heat Transfer Characteristics of Miniature Heat Pipe with Sintered Porous Media	1015
P. Sakulchangsatjatai, N. Thuchayapong, P. Terdtoon and N. Sangsirakoup	1015
Thermomecanical Response of LNG Concrete Tank to Cryogenic Temperatures L. Dahmani and R. MEHADDENE	1021
Electrical Properties of Nanostructured Titanium Dioxide Thin Films Prepared by Sol-Gel	
Spin-Coating Method M.N. Asiah, M.Z. Basri and M. Rusop	1027
Thermal Shock Damage Evaluation of Porous Refractory by Finite Element Method	1027
C. Wang and Y.S. Jiand	1032
The Synthesis of VACNTs Grown on Mg <sub>x</sub> Zn <sub>1-x</sub> O (0	
S. Muhamad, S. Abu Bakar, R. Ahmad, A. Ahmad, Y.M. Siran, S.A.M. Rejab, A.J. Asis, S. Tahirruddin, S. Abdullah and M. Rusop	1038
Synthesis of Supergrowth VACNTs and Nanostructured ZnO by Immerse Method	
S. Muhamad, S. Abu Bakar, M. Zainizan Sahdan, A. Ahmad, Y.M. Siran, S.A.M. Rejab, A.J. Asis, S. Tahirruddin, S. Abdullah and M. Rusop	1044
Experimental Investigation of Gas-Heavy Oil Molecular Diffusion Coefficient in Porous Media: Experimental Results for CO <sub>2</sub> in Iranian Crudes	1040
N. Mirjordavi, M. Kazemeini, R. Kharrat, M.H. Ghazanfari and A. Salehi  Characterization of Interaction I even in I. Mo. V. (X = Nb. 7r.) and II. Nb. 7r. vs. Al	1049
Characterization of Interaction Layer in U-Mo-X (X = Nb, Zr) and U-Nb-Zr vs. Al Diffusion Couples Annealed at 600°C for 10 Hours	
A. Ewh, E. Perez, D.D. Keiser and Y.H. Sohn	1055
Cocoa Husk Waste Mucilage as New Flow Improver in Pipelines H.A. Abdulbari, K.H. Hamad and R.B. Mohd Yunus	1063
Optimization of Dry and Wet Heat Transfer through Textile Structures	
M.J. Geraldes, L. Hes, M. de Araújo, N. Belino and M. Nunes	1068

Design of Multifunctional Truss-Like Periodic Materials Using a Global-Local Optimization Method	
P.A. Muñoz-Rojas, M.A. Luersen, T.A. Carniel and E. Bertoti	1073
Two Phase Dynamic Model for Gas Phase Propylene Copolymerization in Fluidized Bed Reactor	1070
A. Shamiri, M.A. Hussain and F.S. Mjalli Rational Design of TiO <sub>2</sub> -Based Anode Materials Using Computer Modelling	1079
M.V. Koudriachova	1085
Diffusion and Electrophoretic Transport of DNA Polymers in Microfluidic Channels Made of PDMS  R. Sczech, S. Howitz and M. Mertig	1091
Review of Several Studies on High Temperature Oxidation Behaviour and Mechanism of Austenitic Stainless Steels H. Fujikawa	1097
Hydrogen-Induced Superabundant Vacancies in Metals: Implication for Electrodeposition Y. Fukai	1106
Modern Models of Grain Boundary Diffusion V.V. Popov	1116
Growth of Multi-Shaped Zinc Oxide Nanostructures Using C-Axis Oriented Zinc Oxide Thin Film as a Seeded Catalyst via Hydrothermal Aqueous Chemical Growth Method M.H. Mamat, Z. Khusaimi and M.M. Rusop	1126
Optical Properties of Nanostructured Zinc Oxides Deposited on Silicon Substrates M.H. Mamat, Z. Khusaimi and M.M. Rusop	1132
Optimisation of Envelope Insulation for the Retrofit of an Educational Building N.M.M. Ramos, I.M. Ribeiro, P. Santos, J.M.P.Q. Delgado and V.P. de Freitas	1137
Cold-Set Whey Protein Isolate Gels: The Influence of Aggregates Concentration on Viscoelastic Properties	1110
M. Vázquez da Silva and J.M.P.Q. Delgado  Water Vapour Adsorption Study in Spherical Particles Packed in a Cylindrical Container	1143
J.M.P.Q. Delgado and M. Vázquez da Silva	1149
Performance and Modelling of Water Vapour Adsorption in Piles of Granules Using a Cylindrical Pore Model	1177
J.M.P.Q. Delgado and M. Vázquez da Silva  Extraction of Useful Food and Cosmetic Ingredients of Vegetable Origin	1155
M. Vázquez da Silva and J.M.P.Q. Delgado	1161
Simulation and Modeling of Turbulent Plasma Jet Based on Axisymetric LBGK Model R. Djebali, M. El Ganaoui, B. Pateyron and H. Sammouda	1167
Soil Water Characteristic Curve for a Granite Residual Soil: Experimental and Numerical Results	
A. Topa Gomes, A. Viana Da Fonseca and A. Silva Cardoso	1172
Experimental Investigation of the Evaporation and Stability of a Meniscus in a Flat Microchannel	
S. Harmand, K. Sefiane, R. Bennacer and N. Lancial	1178
Diffusion and Ionic Conduction in Soda-Lime Silicate Glasses and in Alkali Borate Glasses H. Mehrer	1184
Mathematical Model of Forecasting the Iron Ore Materials and Coke Quality Indicators A.N. Dmitriev, Y.A. Chesnokov, G.Y. Arzhadeeva and Y.P. Lazebnaya	1198
Mathemathical Model of Powder Sintering with Nano- and Microadditives I.E. Ignatiev, V.I. Bulanov and A.N. Dmitriev	1204
Investigation of Grain Boundary Diffusion in Thin Films by SNMS Technique D.L. Beke, A. Lakatos, G. Erdélyi, A. Makovecz, G.A. Langer, L. Daróczi, K. Vad and A. Csik	1208
Diffusion of Charged Species in Metal Oxides and Polymeric Solids Using AC Impedance Technique  N. Managularman, R. Busdi, N.D. A. Agiz, L. Widerti Zeinstein and Z. Oamen	1017
N. Kamarulzaman, R. Rusdi, N.D.A. Aziz, L. Widarti Zainudin and Z. Osman Comparison of Ionic Diffusion in Sol-Gel Derived Micron and Nano LiTaO <sub>3</sub>	1216
N. Kâmarulzaman, L. Widarti Zainudin, R. Yahaya Subban and Z. Osman	1222
Oxygen Diffusion in an Nb-Ta Alloy Measured by Mechanical Spectroscopy C.R. Grandini, L.H. de Almeida and D. Rodrigues Júnior	1228

Mechanical Spectroscopy Study on Cu <sub>53.5</sub> Zr <sub>42</sub> Al <sub>4.5</sub> Alloy	
O. Florêncio, P.S. Silva, F.H. De Sá, P.W.B. Marques, J.A. Muñoz Chaves, M. Martins Garcia,	
L.C. Rodriguez Aliaga and W.J. Botta Filho	1233
Enhanced UHMWPE Reinforced with MWCNT through Mechanical Ball-Milling	
A. Fonseca, S. Kanagaraj, M.S.A. Oliveira and J.A.O. Simões	1238

Home Journal Rankings Country Rankings Viz Tools Help About Us

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Media Ranking in Switzerland	Condensed Matter		
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	Radiation		
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Journals	10120386, 16629507	1996-2022	Homepage
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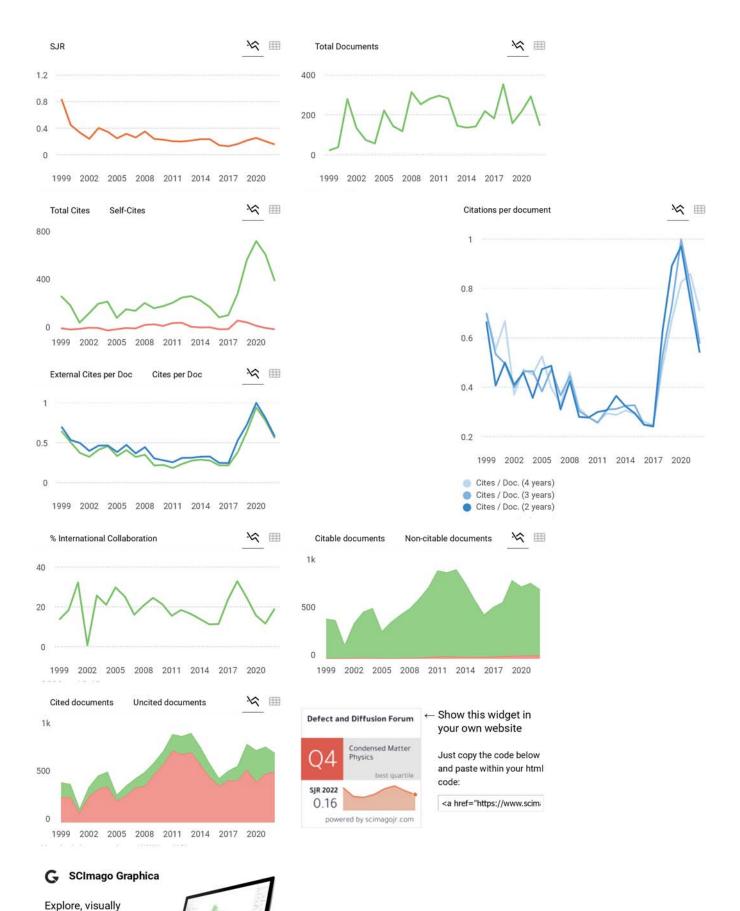
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Best Regards, SCImago Team

### A Andrei Iliescu 3 years ago

Dear Sir,

Would you be so kind to tell me about the Impact Factor between 2017-2020

Thanking you in advance,

Best regards,

Prof.Andrei Iliescu

reply



## Melanie Ortiz 3 years ago

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