



Corrigendum

Corrigendum to “Direct fabrication of crystalline hydroxyapatite coating on zirconium by single-step plasma electrolytic oxidation process” [Surf. Coat. Technol. (2016) 74–79]

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The article entitled ‘Direct fabrication of crystalline hydroxyapatite coating on zirconium by single-step plasma electrolytic oxidation process’ (Surface and Coatings Technology, 301, 74–79 2016) was a subject of “fabrication” complaint. It was claimed that single step HA synthesis was not possible under given experimental conditions and we were accused of fabricating the results of the published article. We have repeated the experimental studies under the observation and in the witness of a scientific committee and the results obtained in the repeated experiments clearly showed that the hydroxyapatite (HA) phase was obtained by the PEO method in a single step PEO process according to XRD and FTIR results as reported in the article. Although, the scientific committee decided that ‘fabrication’ was not committed, the errors found in the published article according to the new results and evaluation must be corrected.

We are sorry for these unintentional errors and would kindly ask you to publish a Corrigendum/Errata by considering the following points;

1. The name of the chemical used for electrolyte preparation was mistyped. In the article the chemical formula was typed as “Ca(CH₃COO)₂ × 5H₂O”. But, the correct formula should have been typed as “Ca(CH₃COO)₂ × H₂O”.
2. The number of the JCPDS card for XRD peak analysis was mistyped. The card number was typed as “Hydroxyapatite-HA JCPDS card no 009-06423” in the published article. But, the correct card number should have been typed as “Hydroxyapatite-HA JCPDS card no: 009-0432”.
3. The wetting angle was measured as 42°, 65° and 79° on a new sample using KSV-CAM 200 Contact Angle Meter though the wetting angle (Rame-Hart Contact Angle Goniometer) was reported as 36 ± 2.8° in the article.
4. Some of the surface SEM images obtained from on new samples were not exactly the same as the images reported in the article (Fig. 3 in the article). The typical SEM micrographs of the new

samples are given in Fig. E1 together with former SEM images (Fig. E2). Fig. E1–b show that the SEM micrograph of the Sample A is not the same as the typical SEM micrograph reported in the article. Although the SEM image of Sample B obtained from higher magnification (Fig. E1d) is similar to the typical SEM image reported in the article; the image with low magnification is different. The SEM micrographs of the Samples C and D (Fig. E1e–j) are typical images reported in the article. Furthermore, the needle-like features that were given as typical in the article are clearly seen on SEM micrographs of sample C and D (Fig. E1e–j) though these features are not seen on the micrographs of sample A and B (Fig. E1a–d). These differences on the SEM images were attributed to the typical inhomogeneous surface morphology of PEO coatings.

5. The functional chemical groups in the coating were examined by FTIR for the spectrum range of 500–4000 cm^{−1}. The presence of PO₄^{3−} and CO₃^{2−} peaks were confirmed for the new samples though these peaks obtained from the new samples are relatively wider and shorter. Both spectrums are shown in the same graph (Fig. E3). Although the spectrums are not identical in terms of peak intensities, both FTIR results confirmed the formation of HA on the surface of Zr substrate with the presence of PO₄^{3−} and CO₃^{2−}.
6. XRD diffraction patterns of new samples obtained from 3 different XRD devices were examined and a typical XRD pattern is given in Fig. E4a. The phase composition of the samples obtained from all examinations was consistent. According to the new XRD analysis, the presence of 3 phases of Ca_{0.15}O_{1.85}Zr_{0.85} (#26-341), m-ZrO₂ (#72-1669), CaZrO₃ (#35-0790) was clearly evident. In addition to these phases, the presence of the HA-Ca₅(PO₄)₃(OH) (#009-0432) phase is also defined according to the result of new XRD analysis. However, the presence of CaZrO₃ was not identified in the previous XRD pattern given in the published article. So, the XRD pattern (Fig. 1 in the article) was also revised and given in Fig. E4b. It should be noted that although the distinguishing peaks of CaZrO₃ (2θ = 22.115°, 31.068°, 37.295° and 37.437°) are clear to be

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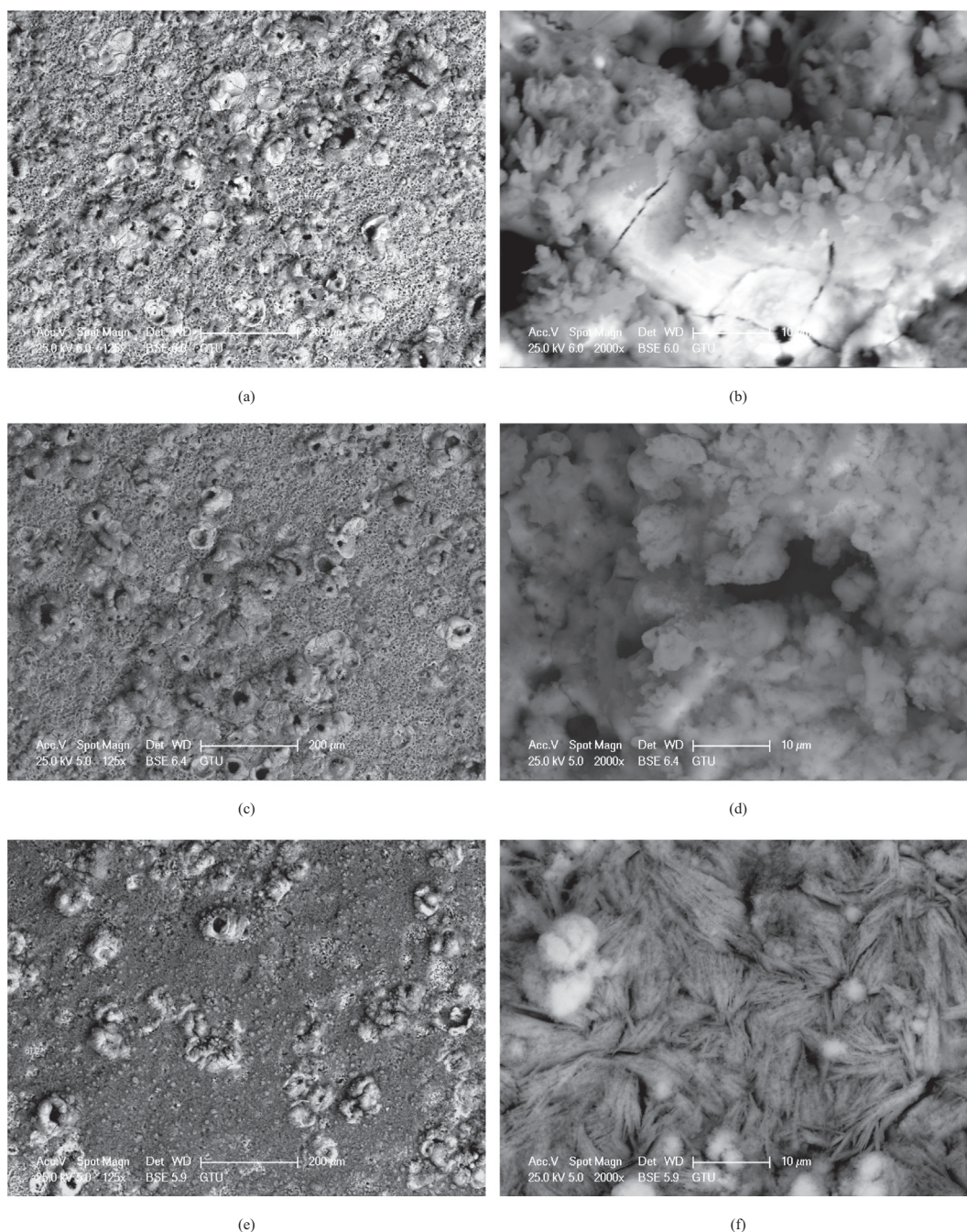


Fig. E1. Surface SEM micrographs of PEO coated pure zirconium at different magnifications of the samples produced in the repeated tests under the same experimental conditions of the article, (a) Sample A-125 \times , (b) Sample A-2000 \times , (c) Sample B-125 \times , (d) Sample B-2000 \times , (e) Sample C-125 \times , (f) Sample C-2000 \times , (g) Sample C-10000 \times , (h) Sample C-50000 \times , (i) Sample D-2000 \times , (j) Sample D-5000 \times .

detected for the new XRD pattern, they were too weak to be detected in the pattern of the article given previously. Thus, in the article, the coinciding peaks of CaZrO_3 and HA phases were misidentified as peaks only belonging to the HA phase. But this was not absolutely correct for the coinciding XRD peaks of CaZrO_3 and HA phases since it is difficult to differentiate both phases from these coinciding XRD peaks. In this case, it will be useful to examine the 2θ values of peaks for each phase of HA and CaZrO_3 to determine the XRD peaks which do not coincide with the XRD peaks of other phases. Thus, the XRD peaks at $2\theta = 22.155^\circ$, 31.068° , 37.295° and 37.437° are distinguishing peaks for CaZrO_3 while the XRD peaks at $2\theta = 25.879^\circ$, 32.902° and 46.712° are distinguishing peaks for HA in the XRD patterns of both examinations. So these XRD peaks have conclusive

evidence to distinguish CaZrO_3 and HA phases from each other. Although both examinations confirm that HA phase can be synthesized on Zr in a single step PEO process, the coinciding XRD peaks of CaZrO_3 and HA phases with higher intensity and sharpness misled us to define HA phase in higher quantities and in higher crystallinity in the coating than they were in reality in XRD pattern given in the article (Fig. 1 in the article).

P.S. Please see an additional note at the end of this document.

An additional short note to the kind attention of the Editor:

A study on the topic of our article, with the details given below was presented orally. We could not mention this study in our article (our article was accessible online on 24th December 2015). Because, neither

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