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Degradation Effect on the Flexural Strength & Micro-Hardness of IPS e-max Laminated Ceramics

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***Abstract* – Ceramic chips play an important role in dentistry when used in a fixed dental damage due to aesthetic, as well as the growing importance and enormity that bring these chips to beauty, and the beauty of the lifestyle imposed by society. These Chips are manufactured in two different ways: (i) IPS e-max press & (ii) IPS e-max cad. The objective of the present work is to study the degradation effect of the IPS e-max laminated ceramic properties as a function of flexural strength and micro-hardness for two different routes of manufacturing the material - IPS e-max press & IPS e-max cad. For the aim of testing the nominated samples an experimental work designed to consist of sixty samples manufactured in two different ways and divided by its type of manufacturing the (IPS e-max press & IPS e-max cad) to two groups which are divided to two subgroups each with fifteen samples assigned to pre- test and post test. The results of the flexural strength and degradation tests show that, using different ways in manufacturing the ceramic chips which classified as: (i) IPS e-max press & (ii) IPS e-max cad resulted in differences in the measured characteristics that affecting the aesthetics of these chips before and after the test. The analysis of the obtained results using the SSPS tools show that the differences are not statistically significant on the measured characteristics of the manufacturing route of either IPS e-max press or IPS e- max cad. However, it is recommended the use of the ceramic chips manufactured using IPS e-max cad route due to its better degradation resistance and mechanical properties.**

***Keywords* – IPS e-max Laminated Ceramics, Flexural Strength, Degradation Effect, Micro-Hardness.**

I. INTRODUCTION

The use of IPS e-max laminated ceramic material as teeth restorations proven to be a durable and aesthetic restorative substance for teeth cures in the front area of the mouth (labial surface of tooth). IPS e-max laminated ceramic in cosmetic dentistry has an important role due to its aesthetic appearance, as well as increasingly important because of the great appeal of the media and beauty style imposed by the society itself [1 & 2]. Consequently, the patients demand is also increasing with regard to the quality of restoration, especially those involving the anterior restorations, but should not be limited to restrict to restore shape and function of the teeth. Accordingly, the ability to recreate a new smile should adapt to the patient's lifestyle, function, and social position, as well as highlight the aesthetic features.

IPS e-max cad recorded less weight loss after being subjected to corrosion test than IPS e-max press. The method of construction affected the flexural strength & micro-hardness of ceramic as machinable ceramic (IPS e-max cad) recorded significant higher data than pressable ceramic (IPS e-max press [3 & 4]. Show in their study the use of glaze layer following polishing of ceramic had significantly improved surface smoothness of ceramic [5]. IPS e-max exhibits significantly higher values in fracture load, flexural strength and hardness [6 & 7]. Lithium disilicate has excellent optical properties, high mechanical resistance, unique restorative versatility, and different manufacturing techniques; it is no doubt one of the most promising dental materials in the realm of digital dentistry [8].

The aim of this work is to study the degradation effect on the IPS e-max laminated ceramic properties as a fun

-ction of flexural strength and micro-hardness for two different routes of manufacturing the material - IPS e-max press & IPS e-max cad techniques.

II. MATERIALS AND METHODS

2.1. *Materials*

IPS e- max laminated ceramic material manufactured by (ivoclar vivadent) Germany, the material is supplied in the form of press and cad. The main constituent of IPE e-max press is pressed glass-ceramic ingot (lithium disilicate crystals), and the IPE e-max cad is a lithium disilicate glass-ceramic block for the cad technique.

2.1.1. *Samples Preparation*

These laminated ceramic materials were manufactured in two different routes (IPS e-max press, IPS e-max cad) according to the manufacturer's instructions.

2.1.2. *IPS e-max Press*

IPS e-max press (Ivoclar Vivadent) consists of Lithium Disilicate, Pressed Glass ceramic samples were fabricated as the thirty samples of cutting the wax sheet was designed and constructed to allow the fabrication of wax rectangular samples with the intended dimension (18x15x1) mm, according to manufactures' instructions and recommendations.

2.1.3. *IPS e-max Cad*

A lithium disilicate glass-ceramic block is used to cut the IPS e-max cad samples for the CAD technique Thirty samples of cutting the cerec lab block by (hypiont water jet cutting machine made in Japan), rectangular sample (18 x 15 x 1) mm, the IPS e-max cad samples attained their final physical properties, and also desired aesthetic properties, such as color, excellent translucency and brightness.

2.2 *Degradation Test*

Abrasive in dental material causes porosity which lead to weakness of laminated ceramic [9]. In this study the degradation rate was done through determine the samples weight loss (mg/cm2). Fifteen samples were prepared of each type of the two laminated ceramic materials which will be subjected to degradation test. Samples were cut with the dimension of (18x15x1) mm, following the manufacturer's instructions and recommendations. The samples were first washed three times with ethyl alcohol then dried. The samples laminated ceramic were weighed then placed in test glass bottles having the same volume of the 4% acetic acid solution at a temperature of 80°C and kept for 16 hours according to the ISO Standards 6872 for hydrolytic resistance of dental ceramic materials [10 & 11].

The degradation rates of the tested samples immersed in the testing media was determined using the standard mathematical relationship.

$$\text{Degradation Rate} = K\, W/DAT \quad (1)$$

Where: **K**: a constant depended on unites used, **D**: Density of sample, **A**: Area of sample, **T**: Exposure time.

The degradation test was carried out at the laboratories of the Advanced Laboratory of Chemical Analysis, Tajoura, Libya). After completing the degradation test, two samples were taken from each material (before &

after) and prepared for the electron microscope test. The scanning electron microscope used - Japan 2000 model IEO 1430VP. The scanning electron microscope test was carried out at the laboratories of the Libyan Petroleum Institute, Tripoli, Libya). The data collected and the mean of fourteen IPS e-max laminated ceramic samples for the both materials was measured, calculated and analyzed using the statistical tool SPSS [9 & 13].

2.3 *Flexural Strength Test*

Strength laminated ceramic in dental material causes increase resistance of the tested laminated ceramic. Flexural Strength test determining force load by (MPa). The universal testing machine, Instron 4411, Massachusetts was use to testing the samples with machine placed symmetrically over 0.5mm span. Seven samples of each of the materials laminated ceramic subjected to of the (before, after) tested laminated ceramic. Samples were cut with the dimension (18x15x1) mm following the manufacturer's instructions and recommendations. Using the standard ISO 6872: 2009 the samples were tested in a three-point flexure with a crosshead speed of a cross head loading rate of 0.5mm /min. The flexural strength was calculated according to equations developed to calculate the fracture stress using the formula,

$$\sigma_f = 3F_1 / 2wh2 \quad (2)$$

Where:

σ_f: is the fracture load, F_1: the roller span (12mm), **W**: the width, h: the height of the bar.

The probability of fracture of different materials was studied using the Weibull cumulative distribution function. Values were analyzed with the following equation [9]:

$$\text{Fracture probability } F = 1\text{-e-VE } (\sigma/ \sigma\ 0)\ m \quad (3)$$

Where $\boldsymbol{\sigma}$: the fracture stress, **VE**: the effective volume, $\boldsymbol{\sigma_0}$: the characteristic strength (the strength occurring at a 63. 2% probability of failure) and **m**: the Weibull modulus. The flexural strength test was done (carried out at the laboratories of the high vocational center of casting, sidi-Assayah, Libya). The data collected and the mean of seven IPS e-max laminated ceramic samples (before, after) for the both two materials was measured, calculated and analyzed using SPSS [13].

2.4 *Micro-hardness Test*

Micro-hardness laminated ceramic in dental material lead to increase resistance susceptibility of fracture of the tested laminated ceramic. Micro-hardness test determining the average micro Vickers Hardness Number (HV) was conducted on seven samples of each group of the laminated ceramic material subjected to (before, after) tested laminated ceramic. Samples were cut with the dimension (18x15x1) mm, following the manufacturer's instructions and recommendations. Using the standard relation according to the international standard ISO 6872: 2009 the surface hardness was measured using digital micro Vickers hardness tester (model microbul1000-dn testing instrument co ltd. Turkey). The tested sample was mounted in the horizontal stage of the tester then the indenter was lowered under a load of 1kg for 10 seconds. The micro-hardness test was done at (carried out at the laboratories of the high vocational center of casting, sidi-Assayah, Libya). The data collected and the mean of seven IPS e-max laminated ceramic samples of each group was measured (before, after), calculated and analyzed using suitable statistical method [9 & 13].

III. RESULTS AND DISCUSSION

The results of the experimental work of this study, degradation effect on the IPS e-max laminated ceramic properties as a function of flexural strength and micro-hardness for two different routes of manufacturing the material – IPS e-max press & IPS e-max cad techniques will be discussed. Sixty samples were made of IPS e-max press & IPS e-max cad and divided into two groups of (30 samples each) according to the type of laminate ceramics: IPS e-max press & IPS e-max cad samples were cut with the dimension (18x15x1) mm, following the manufacturer's instructions and recommendations. The 1mm thickness used for the samples is the thickness indicated for the front area of the mouth for fixed restorations. In the Cerec in-lab 3d system which used in this study is the latest addition of Sirona cad/cam product line. Its advanced software allows for broad range of indications: crown copings, multi-unit bridge frameworks, inlays, onlays and fully contoured crowns out of single solid blocks. [12].

As it stated two types of laminate ceramics tests planned to be tested. These types named IPS e-max Press and IPS e-max Cad. Number of samples prepared for this purpose and divided into two groups according to the type of laminate ceramics. Two subgroups were generated from each group. Subgroup one is not subjected to degradation while subgroup two was subjected to degradation test. Finally, each subgroup divided into two classes according to the type of test (flexural strength and micro-hardness). Before further conducting of data analysis, a set of questions and hypotheses needed to be fulfilled. In this study there are several hypotheses and expectation could be stated as follows:

A. There will be a significant difference between the two tested laminate ceramics as regard weight loss; therefore, hypothesis one formulated as: H1: IPS e-max cad recorded less weight loss than IPS e-max press.

B. There will be a significant difference between the two tested laminate ceramics as regard the flexural strength and the hypotheses are formulated as: H2: IPS e-max cad recorded significant higher strength than IPS e-max press, & *H3*: Degraded samples will be expected to record significant lower flexural strength than non-degraded samples for the two tested laminate ceramics materials groups.

C. Regarding the Vickers microhardness test, the results will show significant difference between the two tested laminate ceramics. H4: IPS e-max Cad will record higher microhardness values than IPS e-max Press, & *H5*: The degraded samples will record no significant microhardness values than non-degraded samples for the two tested laminate ceramics materials groups.

3. 1. *Degradation Test*

The mean value for condition of the (IPS e-max cad) is 8.10, and the mean for condition of the (IPS e-max press) is 35.57. The standard deviation for IPS e-max cad is 4.25 and for IPS e-max press is 12.23. The number of cases in each condition (N) is 14 in Table (5.1). Figure (5.1) graph displays more clarifications about the result of differences.

Table 3.1. Means & standard deviations of weight loss (mg/cm2) of the tested materials after the degradation test.

Material	IPS e-max cad	IPS e-max press
Weight loss	8.10 (4.25)	35.57(12.23)

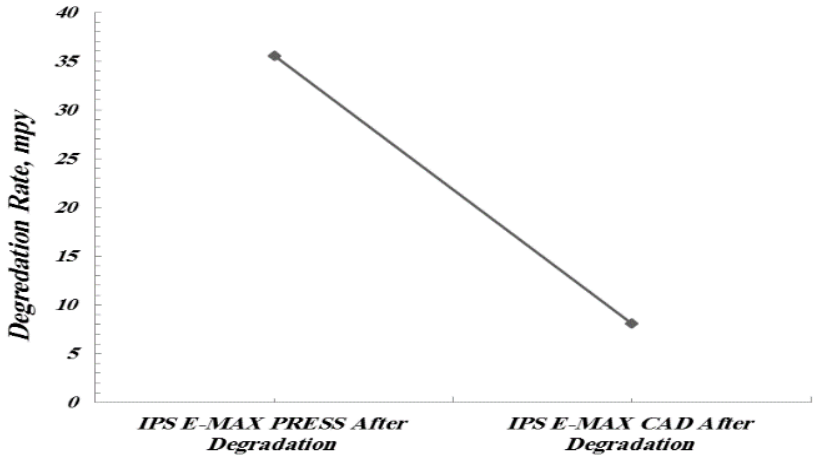

Fig. 3.1. Comparison between the weight loss to degradation test of the tested laminated ceramic.

The degradation test was placed in test glass bottles having the same volume of 4% acetic acid solution at a temperature of 80°C and kept for 16 hours according to the ISO 6872 standards requirements. IPS e-max cad recorded less weight loss than IPS e-max press according to degradation rate, Abrasive in dental material cause porosity which lead to weakness of laminated ceramic results showed. Figure (3.2) shows the scanning electron microscopic.

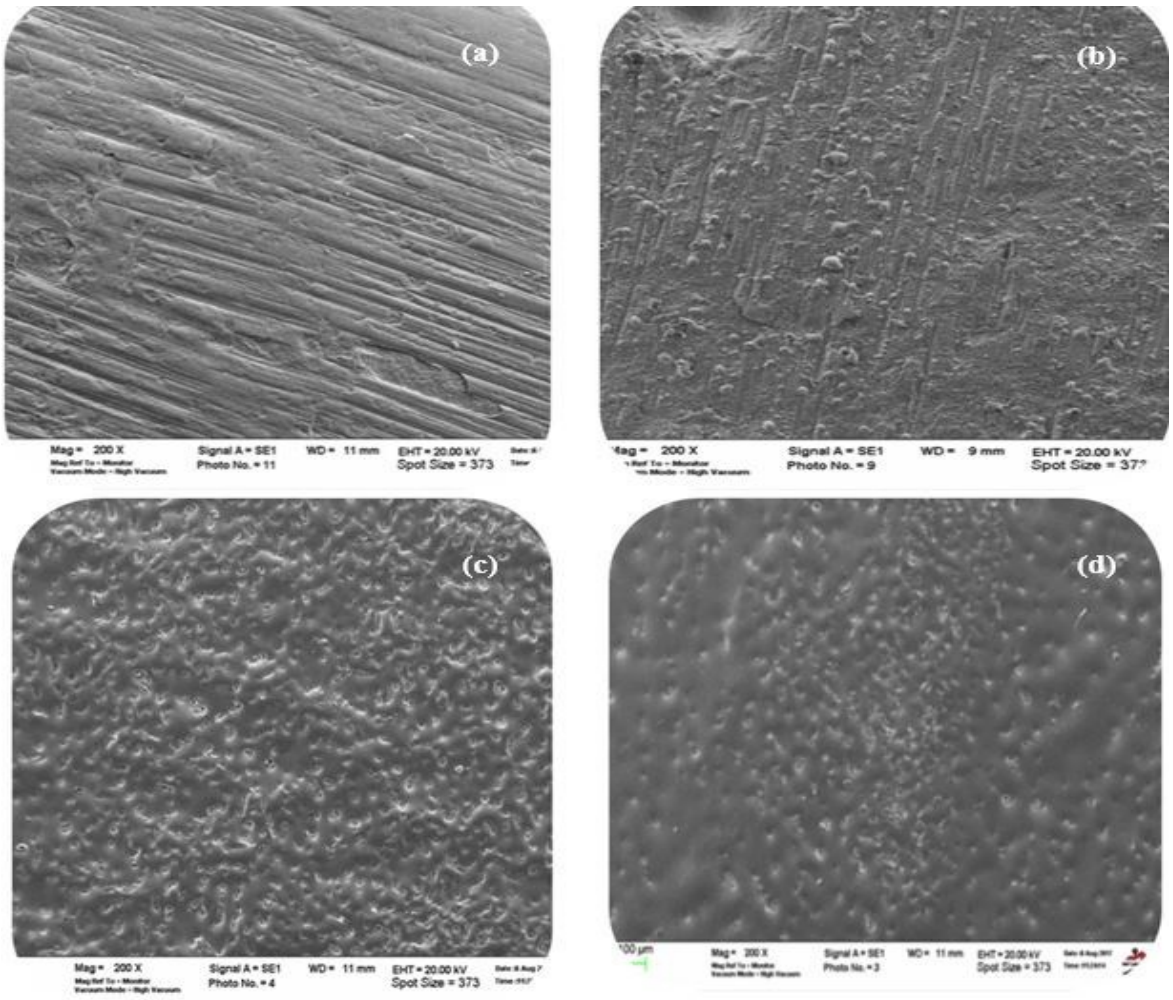

Fig. 3.2. SEM test of the tested laminated Ceramic, (a) IPS e- max press non-degradation (b) IPS e- max press degradation (c) IPS e- max cad non-degradation (d) IPS e- max cad degradation.

3.2 *Flexural Strength*

The degraded samples recorded no significant flexural strength values than non- degraded samples for the two tested laminate ceramics materials of the both groups. Table (3.3) and Figure (3.3) summaries combined clarifications about the result of differences and comparison between the flexural strength of the tested ceramics. Samples were divided into four groups according to their degraded condition (***Group 1***: IPS e-max cad before degraded; ***Group 2***: IPS e-max press before degraded; ***Group 3***: IPS e-max cad after degraded and ***Group 4***: IPS e-max press after degraded). The finding revealed that, there was a statistically significant difference at the $p < .05$ level in values for the four ceramics groups: $F(3, 24) = 65.484$, $p = 0.000$. Post-hoc comparisons using the Tukey HSD test indicated that the mean score for IPS e-max cad before ($M = 364.00$, $SD = 12.90$) was significantly different from IPS e-max cad after ($M = 252.71$, $SD = 38.45$). IPS e-max Press before ($M = 311.43$, $SD = 7.48$) did differ significantly from IPS e-max press after ($M = 210.57$, $SD = 15.04$).

Table 3.2. One-way ANOVA Comparison of effects of degradation test on flexural strength (Mpa) of the tested laminated ceramics at p = 0.000.

Material	Mean	Std. deviations	Mean difference
Group 1 Sample No.7	364.00	12.90	111.29
Group 2 Sample No.7	311	7.48	100.86
Group 3 Sample No.7	252.71	38.45	111.29
Group 4 Sample No.7	210.57	15.04	100.86

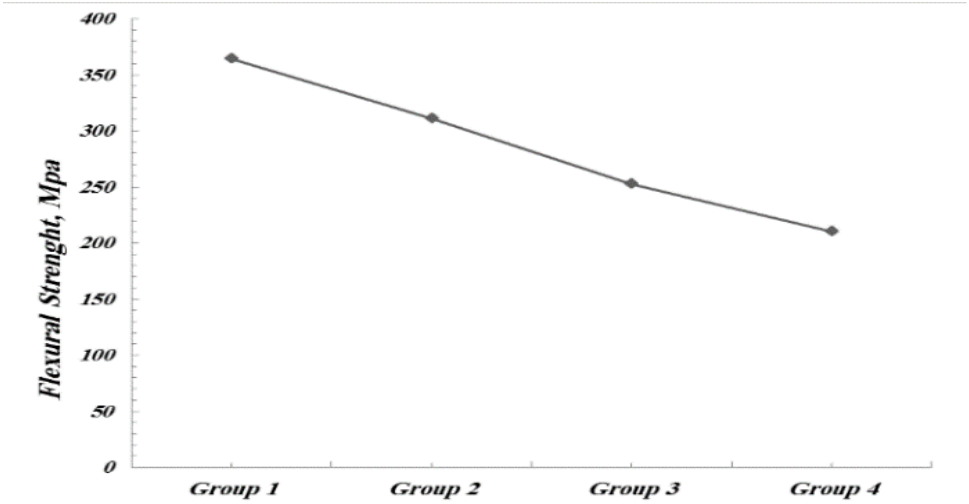

Fig. 3.3. One-way ANOVA Comparison of effects of degradation test on flexural strength of the tested laminated ceramics.

3.3. *Micro-hardness Test*

The degraded samples record no significant micro-hardness values than non- degraded samples for the two tested laminate ceramics. Table (3.3) and Figure (3.4) summaries combined clarifications about the result of

differences comparison between the micro-hardness of the tested ceramics. Samples were divided into four groups according to their degraded condition (***Group 1***: IPS e-max cad before degraded; ***Group 2***: IPS e-max press before degraded; ***Group 3***: IPS e-max cad after degraded and ***Group 4***: IPS e-max press after degraded). The results revealed that, there was a statistically significant difference at the $p < .05$ level in HV values for the four ceramics groups: $F(3, 24) = 118.2$, $p = 0.000$. Post-hoc comparisons using the Tukey HSD test indicated that the mean score for IPS e-max cad before ($M = 568.14$, $SD = 80.04$) was significantly different from IPS e-max cad after ($M = 178.55$, $SD = 12.86$). IPS e-max press before ($M = 405.43$, $SD = 57.08$) did differ significantly from IPS e-max press after ($M = 119.08$, $SD = 19.38$).

Table 3.3. One-way ANOVA Comparison of effects of degradation test on micro-hardness (HV) of the tested laminated ceramics at p = 0.000.

Material	Mean	Std. deviations	Mean difference
Group 1 Sample No.7	568.14	80.04	286.34
Group 2 Sample No.7	405.43	57.08	389.59
Group 3 Sample No.7	178.55	12.86	286.34
Group 4 Sample No.7	119.08	19.38	389.59

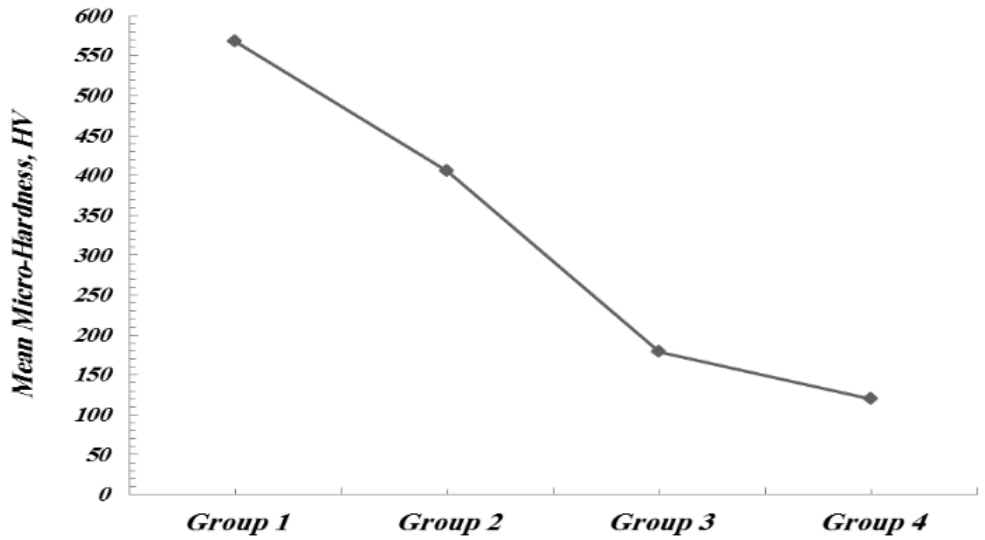

Fig. 3.4. One-way ANOVA Comparison of effects of degradation test on micro-hardness of the tested laminated ceramics.

Figure (3.5. a, b, c & d) show IPS e-max cad recorded higher micro- hardness values. Increase resistance laminated ceramic than IPS e-max press (before degraded). IPS e-max cad recorded higher micro- hardness values which decrease the resistance of laminated increase suability of fracture than IPS e-max press (after degraded).

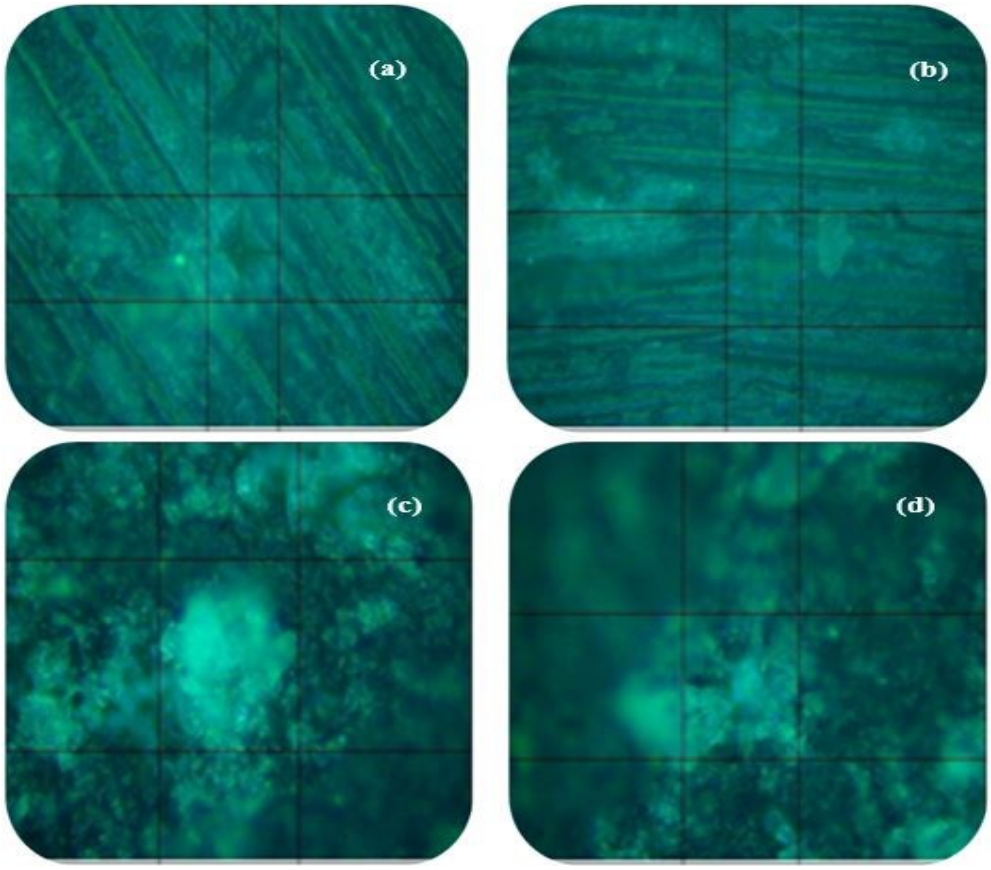

Fig. 3.5. Micro-hardness effect IPS e-max laminated ceramic, (a) IPS e-max press without degradation (b) IPS e-max press degradation, (c) IPS e-max cad without degradation (d) IPS e-max cad degradation.

The experimental work shows that variation in degradation rate affects samples construction: IPS e-max cad recorded less weight loss than IPS e-max press according to the calculated degradation rate. Abrasive in dental material cause porosity which lead to weakness of laminated ceramic. Results show difference between the two tested laminated ceramics flexural strength: IPS e-max cad recorded significant higher strength, and have increased resistance than IPS e-max press (before degraded). IPS e-max cad recorded significant higher strength, decrease resistance of the laminate which increase micro-crake higher than IPS e-max press (after degraded). IPS e-max cad recorded higher micro-hardness values increase resistance laminated ceramic than IPS e-max press (before degraded). IPS e-max cad recorded higher micro-hardness values decrease resistance of laminated increase susceptibility of fracture than IPS e-max press (after degradation).

IV. CONCLUSION

Based on the attained results achieved in this study using the SPSS analytical tool and the test results, this work has led to the following conclusions and remarks:

- The analysis using the SSPS show that the differences are not statistically significant on the measured characteristics of the manufactured techniques of IPS e-max press or IPS e- max cad.
- IPS e-max cad recorded less weight loss than IPS e-max press because of the difference in the manufacturing technology as the IPS e-max cad was subjected to one firing cycles (crystallization & glaze firing) compared to the IPS e-max press which was subjected to two firing cycles (pressing then glazing) of material.

- IPS e-max cad recorded significant higher strength than IPS e-max press due to the difference in the technology of manufacturing.
- Degraded samples recorded significant lower flexural strength than non-degraded samples for the two tested laminate ceramics, and this result affected by the applied heat treatment of the different technology of samples manufacturing.
- IPS e-max cad recorded low micro-hardness values than IPS e-max press due to the difference in the technology of samples construction. In general, based on the measured properties, IPS e-max cad is better than IPS e-max Press.

REFERENCES

[1] C. Hammerle, I. Sailer, A. Thoma, G. Halg & A. Suter, "Dental Ceramics: Essential Aspects for Clinical Practice", Quintessence Publication Co., 2009.
[2] J.M. Powers & J.C. Wataha, "Dental materials: properties and manipulation", 9th edition. Elsevier, St. Louis, 2008.
[3] C. Bergmann & A. Stumph, "Dental Ceramics: Microstructure, properties and Degradation", Springer Science & Business Media, 2013.
[4] C. Mohsen "Corrosion effect on the flexural strength & micro-hardness of IPS e-max ceramics". Published Online June, 2011.
[5] B. Mohammed, B. Afram and Z. Nazar "An Evaluation of the Effect of Different Surface Treatment on Hardness and Smoothness of Pressable Ceramic (*In Vitro Study*)" IOSR Journal of Dental and Medical Sciences, 2015.
[6] D.A. Garber, R.E Goldstein "Porcelain & composite inlays & onlays: esthetic posterior restorations", Quintessence, Chicago, 1994.
[7] A. Albero, A. Pascual, I. Camps, M. Grau-Benitez, "Comparative characterization of a novel cad-cam polymer-infiltrated-ceramic-network" J. Clin. Exp. Dent., 7(4), 2015.
[8] F. Zarone, M. Ferrari, F.G. Mangano, R. Leone, & R. Sorrentino, "Digitally Oriented Materials: Focus on Lithium Disilicate Ceramics", Hindawi Publishing Corporation, International Journal of Dentistry, 2016.
[9] F. Filser, P. Kocher, and F. Weibel, "Reliability and strength of all-ceramic dental restorations fabricated by direct ceramic machining (DCM)", International Journal of Computerized Dentistry, 4, 2001.
[10] K.J. Anusavice, and N.Z Zhang, "Chemical durability of Dicor and Lithia-based glass-ceramics", Dental Materials, 13, 1997.
[11] P. Milleding, A. Wenneberg, S. Alaeddin, S. Karlesson, and E. Simon. "Surface corrosion of dental ceramics in vitro. Biomaterials", 26, 1999.
[12] M. Sekaran, and R. Bougie, "Research methods for business - A skill Building Approach". Wiley, UK 2010.
[13] B.G. Tabachnick, & L.S. Fidell, "Using multivariate statistics" (5th edition). Boston: Pearson Education 2007.
[13] J. Pallant, "A step by step guide to data analysis using SPSS", (4th ed.). Allen & Unwin. Australia 2011.

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