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# Vipac filling test by JNC for pin fabrication of FUJI project

(Technical Document)

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Japan Nuclear Cycle Development Institute

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## (Technical Document)

Yoshiaki Shigetome*

## Abstract

MOX vipac fuel pins will be fabricated in FUJI project to investigate its potential as a fuel for the future commercialized FBR system.

Before the vipac filling test started in PSI, several tests had been conducted to determine the particle size range and preparatory filling tests were performed. In addition, the method to predict the packing fraction was investigated to predict that of the MOX vipac fuel without filling test with MOX particles.

As the result of the investigation, the particle size range was specified as 850-25 μm. The range was divided into polydisperse six fractions and the mixing ratio was deduced in other project of JNC and slightly adjusted for the FUJI project.

Comparison of the particle feeding methods was conducted between the ones with vibration and without vibration, however, it has not been concluded which is better. The effect of particle circularity on the packing fraction was investigated to predict that of MOX. The result shows it would be a promising parameter for evaluation of packing fraction. Since only circularity data of ZrO2 are available at the moment, data of other material are highly desirable to enhance the validity.

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# FUJI プロジェクトにおけるバイパックピン製造のための JNC によるバイパック充填試験

## （技術報告）

重留義明*

## 要旨

FUJI プロジェクトにおいて将来の商用 FBR システムにおけるバイパック燃料のポテンシャルを検討するため、MOX バイパック燃料ピンの製造を行う。PSI において製造試験を実施するに先立ち、使用する顆粒の粒径範囲を定めるための予備試験を実施した。また、MOX 顆粒を用いた充填試験を実施することなく、MOX バイパック燃料の充填率の予測を行うための手法の検討を行った。

試験の結果、粒径範囲は 850-25 μm と定まった。粒度分布は、JNC における他のプロジェクトで見いだした 6 成分の成分比をもとにして、FUJI プロジェクト用に若干修正した。

充填時の粒子投入方法について、ピンを加振する方法としない方法で比較を行ったものの、どちらが優れた方法であるかの結論をすることが出来なかった。MOX バイパック粒子の充填率を予測するために、顆粒の円形度が充填率に及ぼす影響を評価したところ、円形度は充填率予測のための有望なパラメータであることがわかった。現時点では、ジルコニア顆粒の円形度のデータのみであるため、本手法の確立のためには他の物質を用いたデータが望まれる。

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# Vipac filling test by JNC for pin fabrication of FUJI project

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## 1. Introduction

MOX vipac fuel pins will be fabricated in FUJI project. The main purpose of the vipac fuel test in this project is to investigate its potential as a fuel for the future commercialized FBR system. Though the specifications of the vipac pin have not been fixed yet, the goal is to simulate the fuel pins that have been fabricated in RIAR (Research Institute of Atomic Reactors: Russia) [1], [2], because RIAR has the most established vipac fuel technology at the present time.

The number of fractions and the range of the each fraction were determined to imitate the specifications of RIAR and a preliminary experiment was performed at PSI in 2001[3]. The experiment shows the packing fraction was too low. The highest packing fraction obtained in the filling test was 72 %, which was far lower than the targeted value, 80 %. As the large vibration force is necessary to produce a vipac pin with high packing fraction [4], this problem was probably due to shortage of vibration force. This means the vibration force of the vibrator, which is for sphere-pac filling in PSI, is not large enough for vipac filling, therefore the vibrator with larger acceleration force was delivered to PSI from JNC.

Before the filling test in PSI with this vibrator, several tests were conducted to determine the particle size range and preparatory filling tests were performed. In addition, the method to predict the packing fraction was investigated to evaluate that of the MOX vipac pin without performing filling test with MOX particles.

This report shows the recent vipac filling result obtained by JNC.

## 2. Particle size range and weight ratio of each fraction

One of the parameters that had to be specified was particle size range. It should be specified to simulate the range of RIAR that is shown in Table 1[2]. The combination of polydisperse six size fractions has been applied there. Based on this combination, the polydisperse particle size range and weight ratio for this project was determined.

**Table 1 Particle size distribution in RIAR**

| Particle size range (μm) | Weight ratio |
|---|---|
| 800-1000 | 1.4-3.0 % |
| 630-800 | 14.4-22.5 % |
| 400-630 | 18-25.5 % |
| 250-400 | 15.0-24.0 % |
| 100-250 | 19.0-25.0 % |
| < 100 | 14.4-22.5 |

### 2.1. Larger limit of particle size

The range of the largest fraction has been specified as “850-600 μm”, because the sieve size that is close to 800 μm is 850 μm in the both ASTM and JIS (Japanese Industrial Standards) and the particle amount of 800-1000 μm at RIAR is small. It can be considered that the particle amount whose size is between 850-800 μm is large enough to compensate deleting the fraction of “1000-800 μm”, and if it’s not the case, the effect of lacking this small amount of large particle is probably negligible. In addition, as it is desirable to simplify the vipac particle fabrication process, reducing the number of fractions is preferable.

### 2.2. Smaller limit of particle size

It is generally known that the particle mixture with finer particles gives higher packing fraction, then the smaller particle limit should be as small as possible from the standpoint to obtain high packing fraction [4].

The vipac pins for irradiation consist of a fuel region and upper and lower insulator regions, which are filled with spheres with 100 μm diameter. There will be fuel seal discs (FSD) placed between the insulators and fuel column to avoid infiltration of fine fuel particles into insulator regions during fabrication, especially vibro-filling process. The specifications of the cladding and the FSD diameters show the gap width between them can be around 40 μm and there is certain possibility of infiltration.

Therefore we should compromise over the packing fraction and the smaller size limit was determined by the infiltrating potential of fine particles. Several experiments were performed to evaluate the amount of fine particles that can penetrate into the insulator region during vibrocompaction.

The experiments were performed with $ZrO_2$ vipac particles and $HfO_2$ spheres (diameter = 100 μm). The $ZrO_2$ particles and $HfO_2$ spheres were used as dummy vipac fuel particles and dummy insulator spheres respectively.

1) Infiltration test with single fraction

Dummy insulator spheres ($HfO_2$) were fed into the bottom of a pin and a FSD was placed on the insulator. Dummy vipac particles ($ZrO_2$) of single fraction (75-45 or 45-25 μm : 6 g) were fed on the FSD. The pin was vibrated at 500 Hz with 5 G for 5 minutes. After vibration, the insulator spheres were discharged from the pin and sieved by a 75 μm mesh.

The result shows the amount of vipac particles that infiltrated into insulator was quite small (Table 2). This means the amount of vipac particles that infiltrates into insulator is negligible.

**Table 2 Amount of particles that penetrated into insulator**

| Particle range (μm) | Amount of particles (g) |
|---|---|
| 75-45 | not detected |
| 45-25 | 0.009 |

2) Infiltration test with six fractions

Infiltration test was performed also with six fraction particles. Several degrees of frequency (500-2000 Hz) and acceleration (5-50 G) were applied to evaluate the effect of vibration conditions. Mixing ratio of each fraction applied in the tests is shown in Table 3. Particles of 125-75 μm were omitted because the amount of vipac $ZrO_2$ particles that infiltrate into the insulator region was measured by sieving the particles in the insulator region that had initially been composed of only $HfO_2$ spheres with 100 μm diameter before vibration. The results are shown in Table 4 and Table 5.

The results show that the amount of particles that infiltrated into the insulator region was quite small. Therefore it is concluded that the FSD is effective to prevent the particles that are as fine as 25 μm from penetrating into the insulator region.

Accordingly the smaller limit of particles for fuel column has been specified as

25 μm.

**Table 3 Mixing ratios of six fraction particles for infiltration test**

| Range of fraction (μm) | | 850-600 | 600-425 | 425-250 | 250-125 | 75-45 | 45-25 |
|---|---|---|---|---|---|---|---|
| Weight ratio (%) | Frequency test | 50 | 5 | 10 | 5 | 15 | 15 |
| | Acceleration test | 50 | 5 | 10 | 5 | 10 | 20 |

**Table 4 Amount of particles that penetrated into insulator region (frequency test)**

| Frequency (Hz) | 500 | 750 | 1000 | 2000 |
|---|---|---|---|---|
| Acceleration (G) | 10 G | | | |
| Amount of particles (g) | not detected | 0.001 | 0.001 | not detected |

**Table 5 Amount of particles that penetrated into insulator region (acceleration test)**

| Frequency (Hz) | 500 | | | | |
|---|---|---|---|---|---|
| Acceleration (G) | 5 | 10 | 15 | 25 | 50 |
| Amount of particles (g) | not detected | 0.002 | 0.006 | 0.004 | 0.001 |

2.3. Particle size distribution for fuel column

The optimization of the mixing ratio of vipac particles was performed in other JNC project and the mixing ratio shown in Table 6 was deduced by systematical parametric survey [4]. The fraction ranges between the largest and smallest sizes were set as similar as the ones in Table 1. Though the optimized mixing ratio includes 1000-850 μm fraction, the largest fraction in this project has been specified as 850-600 μm, as discussed before. The effect of lacking this fraction makes only small decrease in packing fraction (about 1 % [4]), therefore, the amount of the 1000-850 μm fraction was simply added together with 850-600 μm fraction. Consequently the mixing ratio in Table 7 has been proposed for the vipac fuel of the project.

**Table 6 Deduced mixing ratio as a optimized mixing ratio [4]**

| Particle range (μm) | 1000-850 | 850-600 | 600-425 | 425-250 | 250-106 | 106-45 | 45- |
|---|---|---|---|---|---|---|---|
| Weight ratio (%) | 40.9 | 9.1 | 9.1 | 4.5 | 4.5 | 18.2 | 13.6 |

**Table 7 Mixing ratio proposed for the project**

| Particle range (μm) | 850-600 | 600-425 | 425-250 | 250-106 | 106-45 | 45-25 |
|---|---|---|---|---|---|---|
| Weight ratio (%) | 50.0 | 9.1 | 4.5 | 4.5 | 18.2 | 13.6 |

# 3. Vipac filling test

The filling test in JNC consisted of (1) investigation of particle feeding method and (2) investigation of the effect of particle shape factor on packing fraction. The investigation of the feeding method was to improve the homogeneity of the axial distribution, especially bottom end of the pin. The shape factor was investigated to predict the packing fraction of MOX particles. Since performing a MOX filling test at PSI is practically impossible, it is necessary to predict the packing fraction of MOX vipac fuel without filling test with MOX particles. As the particle shape factor is generally considered to have effect on the packing fraction [5], several trials were performed to evaluate the quantitative effect.

## 3.1. Particle feeding method

### 3.1.1. Inactive filling test with $ZrO_2$

The results of γ-scanning analysis that was performed in the previous vipac filling test at PSI [3] show the density of the bottom end of the column is lower than average column density. It is considered that only large particles exist in the low density region and the cause of this defect is presumed as following mechanism.

When particles are fed into the pin, both friction force from a inner cladding wall and air resistance against larger particles are smaller than those against smaller particles, therefore, the larger particles go down to the bottom of the pin faster than the smaller ones. Then there would be a layer with only large particles at the bottom of the pin before vibration. Particles are supposed to move into the closely packed arrangement during vibration, however, the particles at the bottom end is less mobile because of the weight of the particles that are fed on them and fails to be packed closely.

Filling experiment was performed with a transparent acrylic pin to check this presumption. The mixing ratio of the particles was same as the one in Table 6. The length of the pin was 100 cm and the column length was 80 cm. Though these lengths are longer than the ones for the FUJI project, this extreme experiment is effective to reveal more about the presumption.

After $ZrO_2$ particles fed into the pin, a white layer that indicated the segregation of large particles was observed. The feeding was performed by hand operation. This pin was vibrated for 5 minutes at frequency of 500-1000 Hz (with frequency sweeping at 100 Hz/s) and acceleration of 25 G. There was still the white layer at the end of the pin that indicates the mixing of the large and small particles in this region was not complete.

To avoid this low density region, vibrating a pin while particle feeding was applied and consequently the white layer at the bottom end disappeared. By this method,

particle mixing was gradually completed from the bottom to the top as the progress of feeding.

Table 8 compares the test conditions and packing fractions given by different feeding methods. The vibration time of “feeding with vibration” is equivalent to the feeding time, that is, feeding time is same as vibration time and the vibration was stopped at the same time of the end of feeding. The feeding was performed by hand operation. Though homogeneity of the axial distribution obtained by the feeding with vibration looked better than the other (the homogeneity of the pin was checked by only visual observation from outside of the transparent acrylic pin), difference of the packing fractions between the two was small.

**Table 8 Results of the filling tests by particle feeding with and without vibration**

| Test pin ID | 1 | 2 |
|---|---|---|
| Loading method | Feeding without vibration | Feeding with vibration |
| Pin length (mm) | 1000 | |
| Column length (mm) | 800 | |
| Frequency (Hz) | 500 – 1000 | |
| Acceleration (G) | 25 | |
| Vibration time (min.) | 30 | 43 |
| Packing fraction (%) | 80.8 | 81.0 |

3.1.2. Filling test with $UO_2$

Filling tests with $UO_2$ were performed in order to compare the homogeneity of axial distribution of the pins whose particles were fed with and without vibration. Particle size distribution was same as in Table 6 and the filling conditions and obtained packing fraction are shown in Table 9. The packing fraction of the pin fed with vibration was higher than that of the pin fed without vibration. The axial density distribution was measured by processing their x-ray photos (Fig. 1). From this result the density of the bottom part was improved by particle feeding with vibration, however, there was still low density region in the upper part and the local density in upper part of the pin fed with vibration was even lower than that of the pin fed without vibration. The cause was probably segregation of particles, that is, amount of small particles remained in the bottle for feeding was more than that of large particle, therefore the ratio of (fine particle)/(large particle) came to be higher than the optimum value, though the particles were fed quite carefully.

Some improvement was achieved by feeding, however the homogeneity of the

density distribution was still unsatisfactory. It was difficult to judge which feeding method is better, then both methods were applied in the filling test described hereafter.

**Table 9 Results of the UO2 filling tests by particle feeding with and without vibration**

| Loading method | Feeding without vibration | Feeding with vibration |
|---|---|---|
| Pin length (mm) | 400 | |
| Column length (mm) | 150 | |
| Frequency (Hz) | 500 – 1000 | 400-600 |
| Acceleration (G) | 35 | |
| Vibration time (min.) | 10 | 15 |
| Packing fraction (%) | 79.3 | 81.0 |

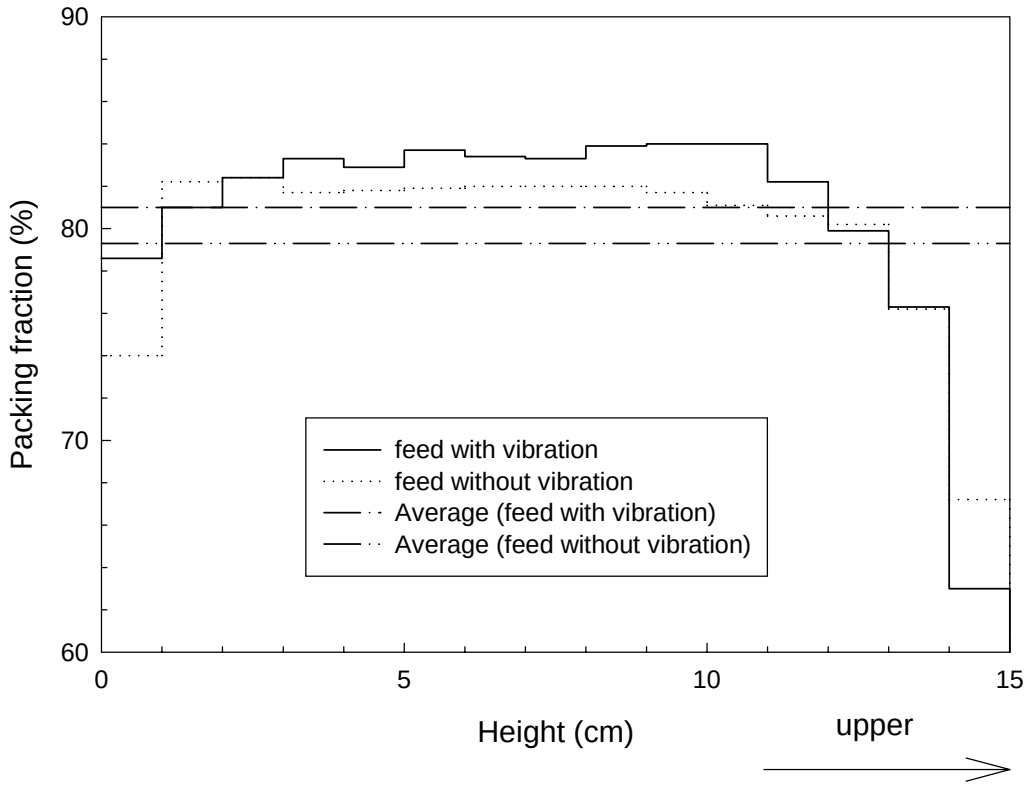



**Fig. 1 Axial density distribution of the pins whose particles were fed with or without vibration**

3.2. Evaluation of shape factor

It is generally considered that higher packing fraction is obtained by rounder particles in vipac filling. $ZrO_2$ particles with three different kinds of roundness were prepared by ball-milling in order to evaluate the effect of roundness on the packing fraction quantitatively. For this purpose, 'circularity' was applied to evaluate roundness of particles.

3.2.1. Evaluation of circularity

The circularity $C$ is defined as

$$C = \frac{r_1}{r_2} (\leq 1.0) \cdots (1)$$

$$r_1 = \sqrt{\frac{S}{\pi}} \cdots (2.a)$$

$$r_2 = \frac{l}{2\pi} \cdots (2.b)$$

$S$ : area of the projected particle image

$l$ : peripheral length of the projected particle image

$r_1$ : radius of the circle whose area is equivalent to the projected particle area

$r_2$ : radius of the circle whose peripheral length is equivalent to the projected particle image.

The $ZrO_2$ particles with three different degrees of roundness were adjusted by ball-milling time (0, 24, 40 hours). The samples are designated as 0H, 24H, and 40H, respectively. Circularity of each sample was evaluated by averaging about 100 particles/sample. Table 10 shows the result of circularity evaluation.

**Table 10 Circularity of $ZrO_2$ samples**

| | Range (μm) | | | | |
|---|---|---|---|---|---|
| | 850-600 | 600-425 | 425-250 | 250-106 | 106-45 |
| 0H | 0.884 | 0.883 | 0.861 | 0.874 | 0.878 |
| 24H | 0.904 | 0.908 | 0.889 | 0.884 | 0.884 |
| 40H | 0.948 | 0.929 | 0.909 | 0.891 | 0.895 |

3.2.2. Pour and tap densities

Pour and tap densities of each sample were measured to investigate the shape factor on packing behavior.

A graduated cylinder with 25 ml volume was prepared and about 60 g of

particles were fed into it. The volume was measured before tapping to calculate the pour density. The cylinder was tapped (dropped from about 60 mm height) for 20 times then the volume was measured again for tap density.

The result of each fraction is shown in Fig. 2. The plot for $c$=1.0 is reference value that is calculated by the equation derived by Ayer [6] that gives the packing fraction of the spheres. The pour and tap densities for mix fraction means the ones with particles composed of six fraction mixed with the ratio shown in Table 7. The circularity for the mixture was calculated by the following equation

$$C_m = \sum_i w_i C_i \quad (3)$$

$w_i$ : weight of $i$th fraction

$c_i$ : circularity of $i$th fraction.

The result indicates the particles with higher circularity gives the higher tap and pour density, therefore the circularity can be regarded as a suitable parameter for evaluation of packing fraction.

The tap and pour densities are same in almost all the fractions except 0H of 250-106 μm and all three fractions of 106-45 μm. This means the particles larger than 250 μm are mobile enough to be closely densified without tapping. On the other hand, the fractions whose pour and tap densities are different are less mobile and require driving force, such as tapping, to be densified. The pour and tap densities of mixture particles are also different. This is probably because they include the smallest fractions that require driving force to be packed closely.

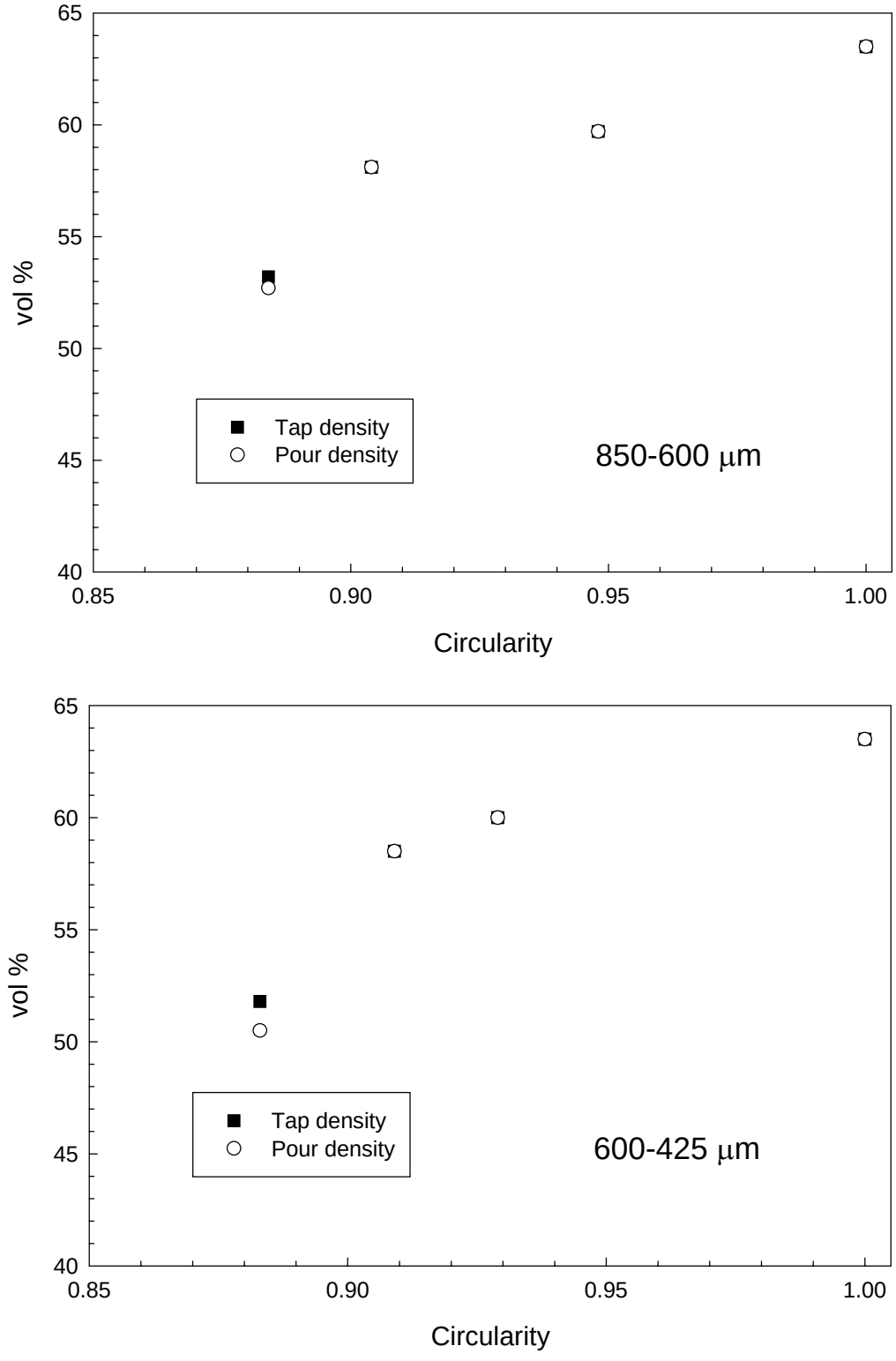
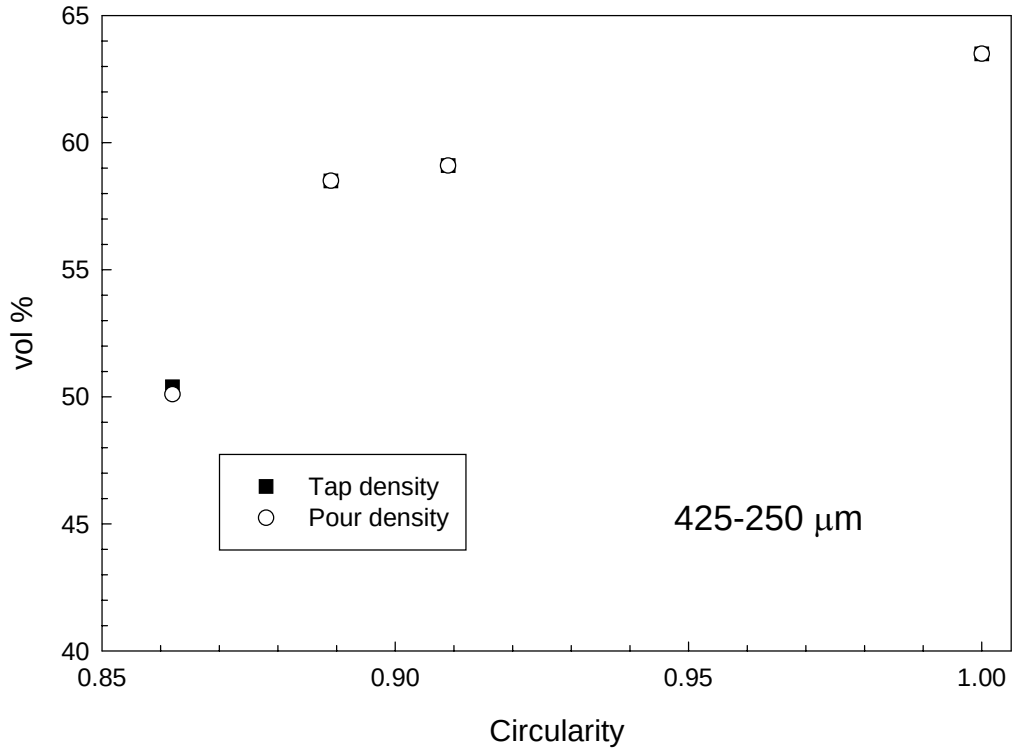



**Fig. 2 Tap and pour density of ZrO2 particles**



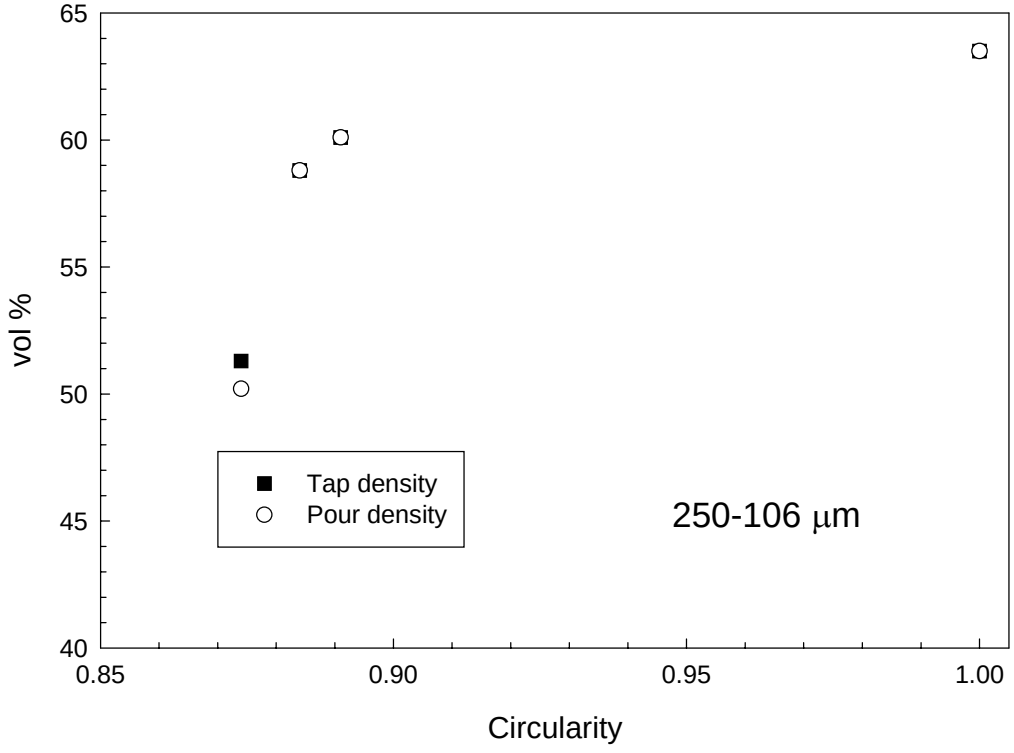
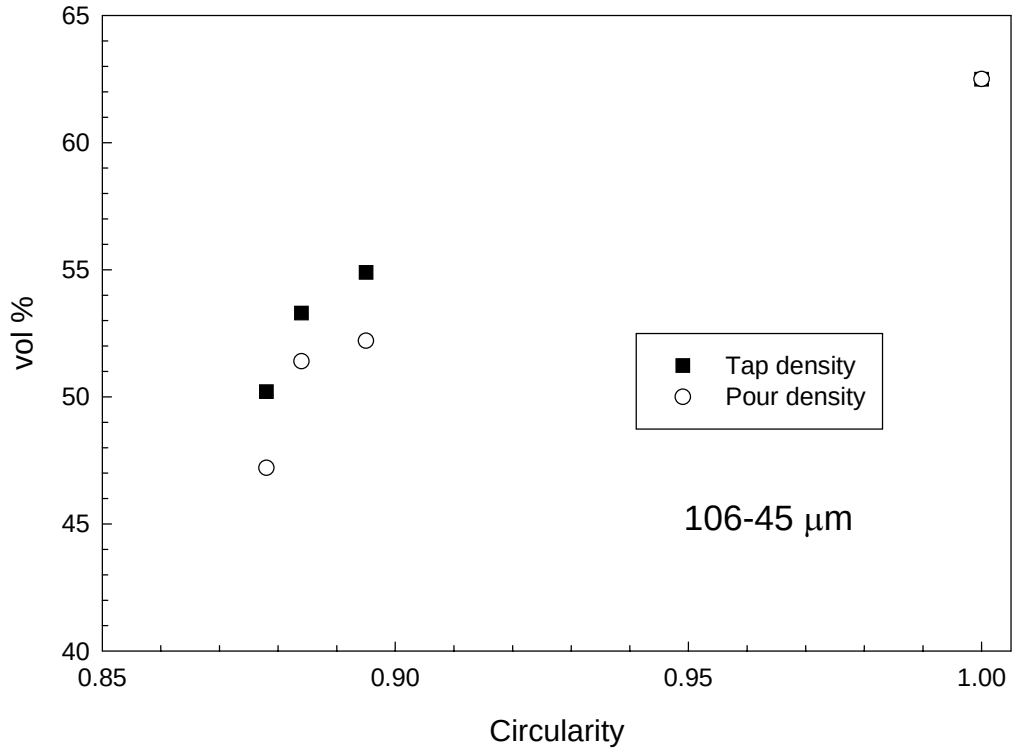




**Fig. 2 (cont.) Tap and pour density of ZrO2 particles**



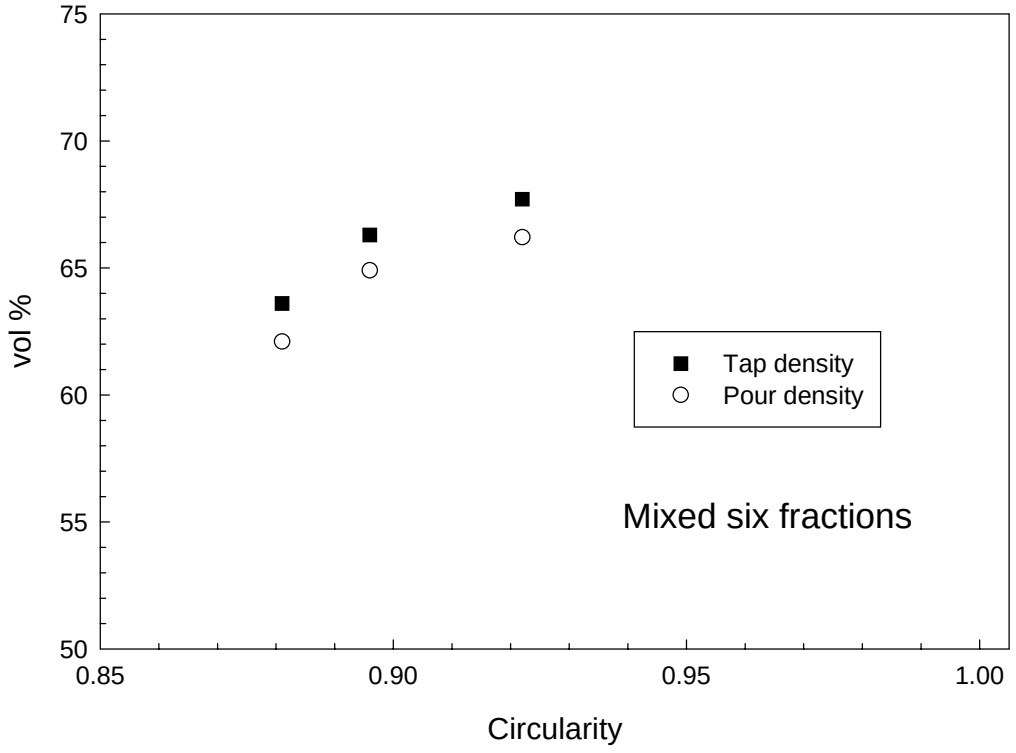




**Fig. 2 (cont.) Tap and pour density of ZrO2 particles**

3.2.3. Effect of circularity on packing fraction

Filling tests were performed to investigate the effect of circularity on the packing fraction of vipac pins. The tests were conducted by the vibrator that was transported to PSI (IMV ; Fig. 3(a)) and the other vibrator (EMIC ; Fig. 3(b)). The EMIC vibrator has feedback control system, but the IMV vibrator doesn't have it, therefore acceleration changes during vibration with frequency sweeping.

In the tests with the IMV vibrator, particles were premixed and fed into a pin without vibration. The tests with the EMIC vibrator were performed for both particle feedings with and without vibration. The feeding time of the test with vibration was about 5 minutes and the packing fraction was measured for three times; after feeding, after 5 minutes vibration, and after additional 10 minutes (total 15 minutes vibration) was applied. The filling conditions and results are shown in Table 11, Fig. 4, and Fig. 5.

The packing fractions given by the feed without vibration (both IMV and EMIC) shows that higher packing fraction is obtained by the particles with higher circularity. The effect of the acceleration was not recognized over 15 G with EMIC, that is, acceleration of 15 G is high enough to relocate $ZrO_2$ particles into the arrangement with the highest packing fraction.

The results given by the feeding with vibration show the highest packing fraction is obtained with the 24H $ZrO_2$ sample, which is inconsistent to the other result. The reason may be the handling of the feed process.

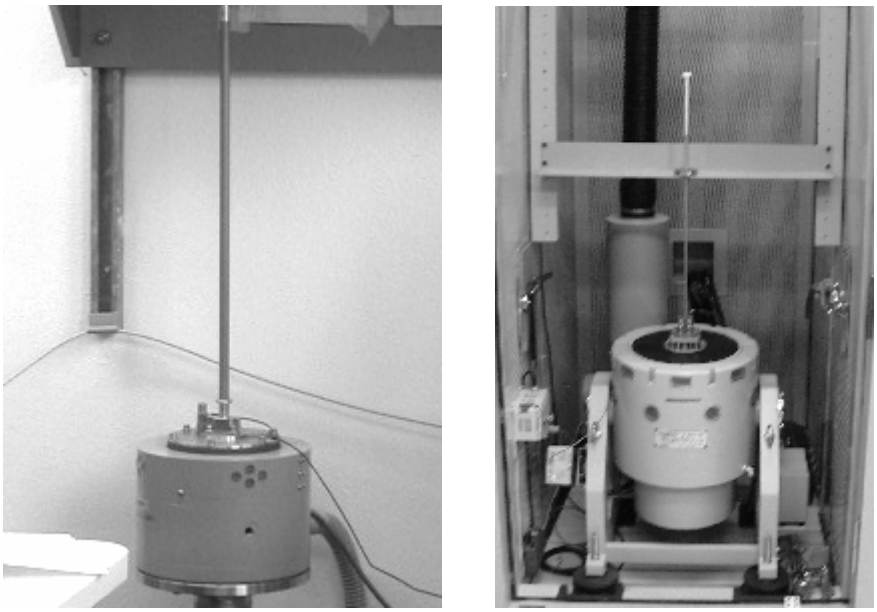
Packing fractions given by feed with vibration is higher than the ones given by without vibration. This is probably due to the homogeneity of the density distribution. The feeding operation of both tests was performed by hand operation, therefore heterogeneous feeding is likely to occur. The heterogeneous feeding was more likely to be recovered in the feeding with vibration than without vibration, that is, feeding with vibration gives more homogeneous axial density distribution and the packing fraction given by feeding with vibration is higher than the one without vibration. However, it is difficult to conclude that feeding with vibration always gives higher packing fraction than without vibration, because measurement of the axial distribution was not performed for these pins.

Although axial distribution of the pin was unknown, the packing fraction shows the tendency that the packing fraction is described as a function of circularity, therefore, the circularity of the particle would be a promising parameter for evaluation of packing fraction. Since only circularity data of $ZrO_2$ are available at the moment, data of other material are highly desirable to enhance the validity.

**Table 11 Filling conditions and results**

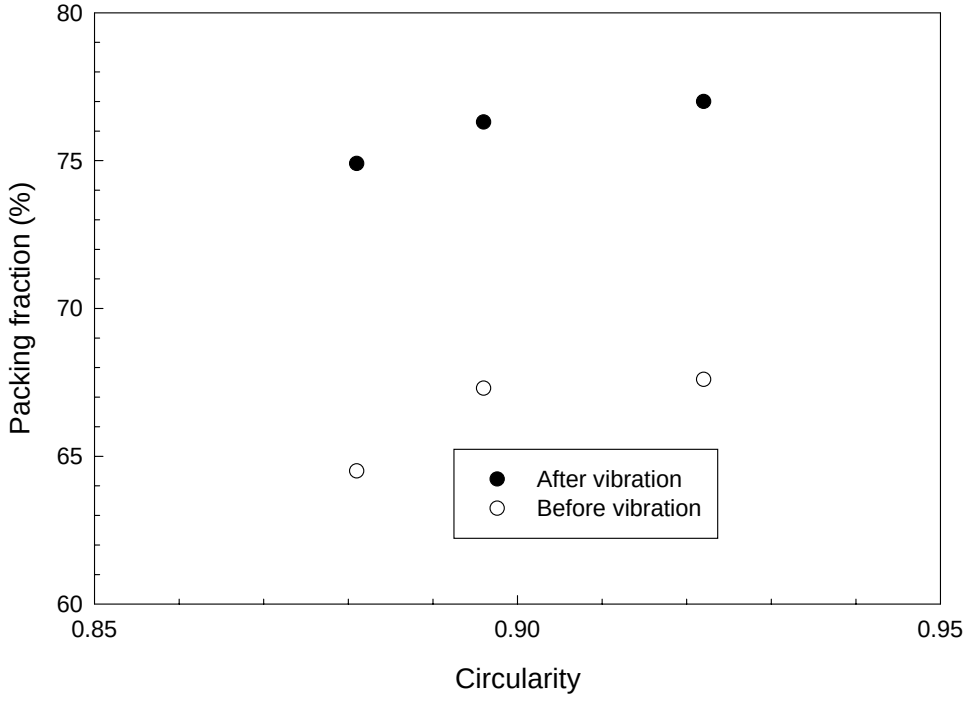
| | | IMV | EMIC | |
|---|---|---|---|---|
| Feeding | | without vibration | without vibration | with vibration |
| Acceleration | | 18 G at 750 Hz | 5, 10, 15, 25 G | 10 G |
| Frequency | | 500-1000 Hz | 500-1000 Hz | 200-400, 400-600, 500-1000 Hz |
| Packing fraction | 0H | 74.9 % | 73.4 % ;(5 G)<br>74.5 % ;(10 G)<br>75.5 % ;(15 G)<br>75.4 % ;(25 G) | 75.1 (76.6) %* ;(200-400 Hz)<br>74.6 (76.9) % ;(400-600 Hz)<br>74.6 (76.6) % ;(500-1000 Hz) |
| | 24H | 76.3 % | 75.6 % ;(5 G)<br>77.1 % ;(10 G)<br>76.7 % ;(15 G)<br>77.1 % ;(25 G) | 76.9 (78.7) % ;(200-400 Hz)<br>77.1 (78.4) % ;(400-600 Hz)<br>77.0 (78.6) % ;(500-1000 Hz) |
| | 40H | 77.0 % | 75.9 % ;(5 G)<br>77.1 % ;(10 G)<br>77.2 % ;(15 G)<br>76.9 % ;(25 G) | 76.6 (78.0) % ;(200-400 Hz)<br>76.7 (78.2) % ;(400-600 Hz)<br>76.9 (77.9) % ;(500-1000 Hz) |

* 75.1 (76.6) % means packing fraction is 75.1 % at the end of feeding and 76.6 % after additional 10 minutes vibration.

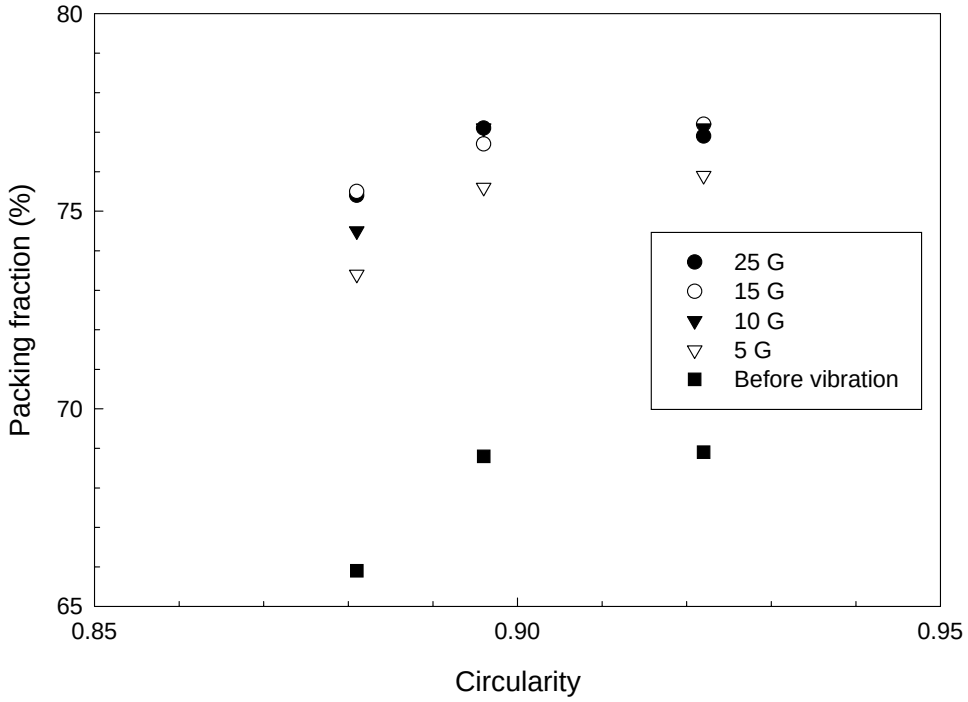


(a) IMV vibrator. (b) EMIC vibrator

**Fig. 3 Images of vibrators**

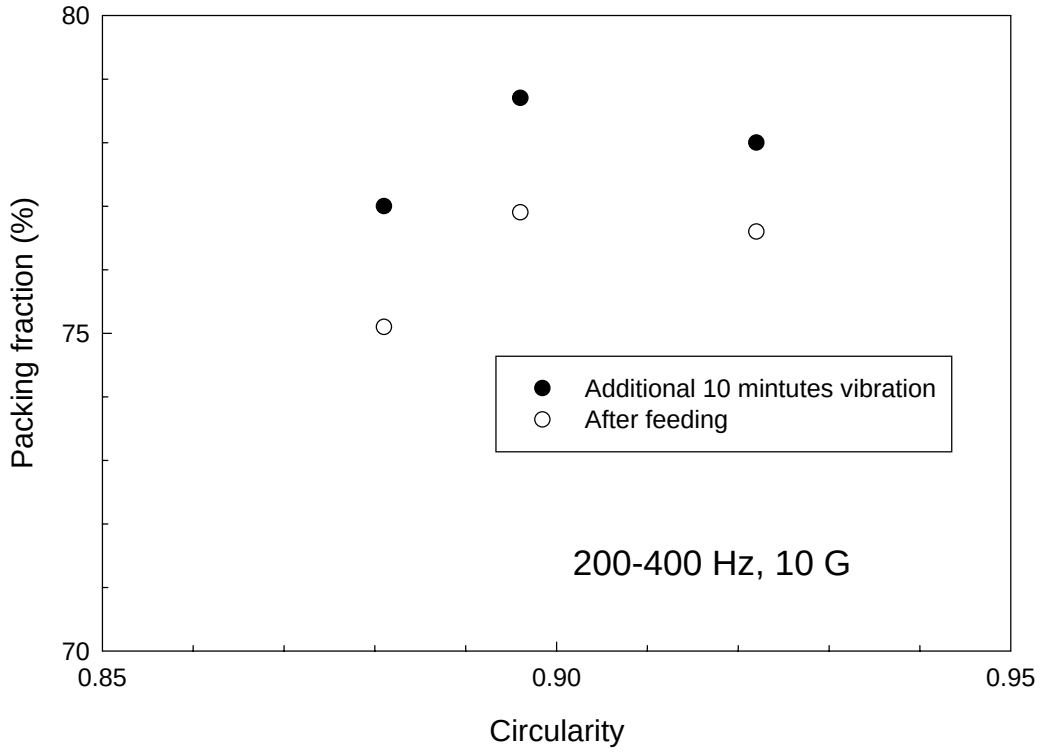



(a) IMV vibrator

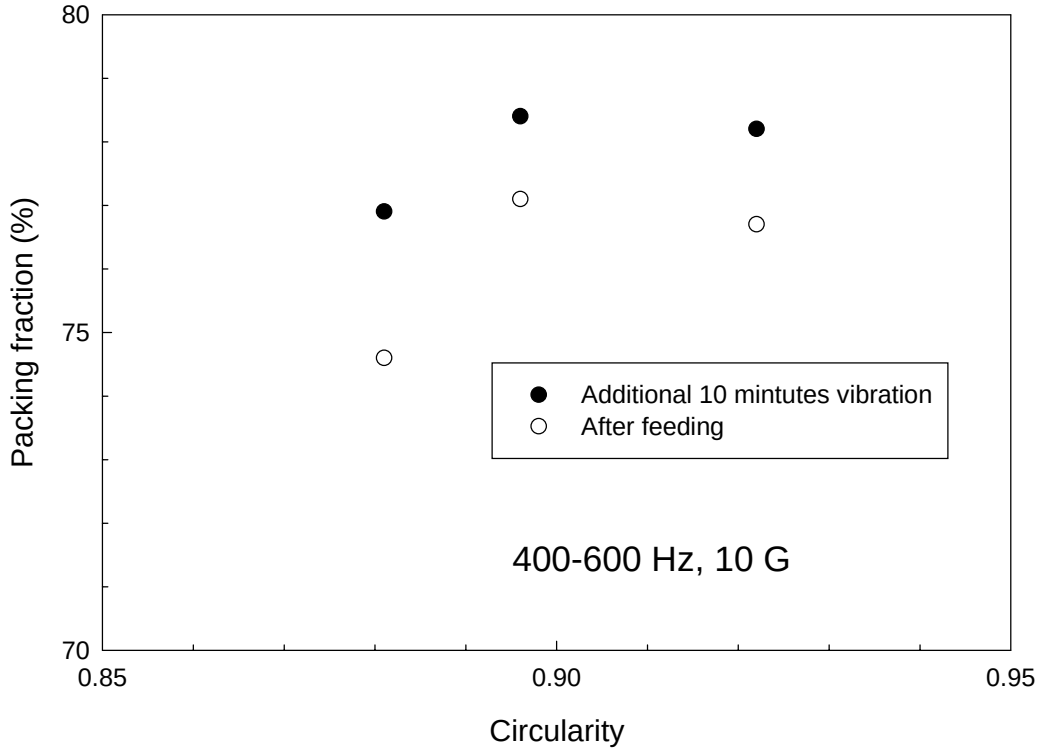



(b) EMIC vibrator

**Fig. 4 Packing fraction obtained by particle feeding without vibration**

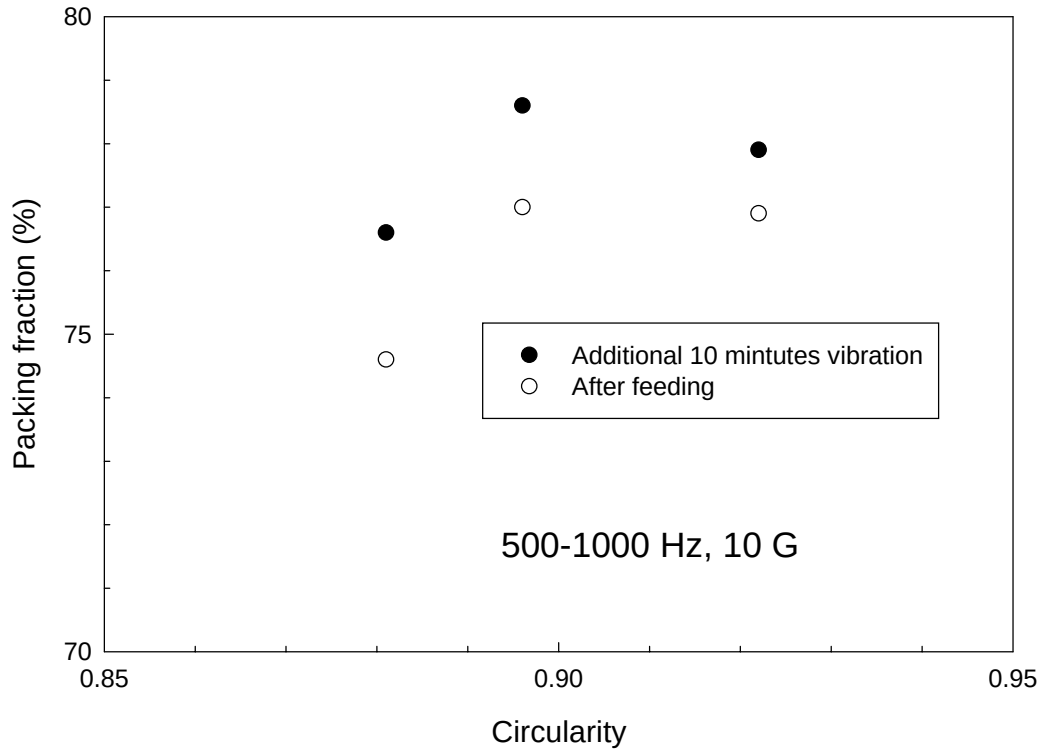



(a) 200-400 Hz




(b) (400-600 Hz)

**Fig. 5 Packing fraction obtained by particle feeding with vibration (EMIC vibrator)**




(c) 500-1000 Hz

**Fig. 5 (cont.) Packing fraction given by particle feeding with vibration (EMIC vibrator)**

## 4. Conclusion

Investigation and filling test for vipac fabrication of FUJI project were performed by JNC. The range of the particle size and size distribution were specified and filling tests were conducted with this distribution.

The particle size range was determined as 850-25 μm. The largest size was defined mainly to simulate the range applied at RIAR [2]. The smallest size was defined by infiltrating behavior of the particles and the test showed the FSD is effective to prevent particles whose size is as small as 25 m from infiltrating into the insulator region. The range was divided into polydisperse six fractions as similar as the fraction ranges at RIAR [2]. The mixing ratio was deduced by systematical parametric survey in other project of JNC and slightly adjusted for the FUJI project.

Comparison of the particle feeding methods was conducted between the one with vibration and without vibration. Feeding with vibration generated higher packing fraction than the one without vibration, however it is difficult to conclude that the feeding with vibration is better, since homogeneity of the axial density distribution was not measured. It is desirable to compare the axial homogeneity of these pins in future.

The effect of the circularity on the packing fraction was investigated to predict the packing fraction of MOX particles without performing a MOX filling test. The packing fraction shows the tendency that it can be described as a function of circularity, therefore the circularity of the particle would be a promising parameter for evaluation of packing fraction. Since only data of $ZrO_2$ are available at the moment, data of other material are highly desirable to enhance the validity.

In the FUJI project, packing fractions of both $ZrO_2$ and MOX particles are supposed to be obtained. They are expected to be valuable data to demonstrate the correlation between circularity and packing fraction.

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