Project: Elimination of TiN Peeling During Exposure to CVD Tungsten Deposition Process Using Designed Experiments

Introduction

Aim of the study

There're tradeoffs among TiN peeling, tungsten film uniformity and tungsten film stress, where uniformity and stress were founded to be controlled by both pressure and H_2/WF_6 ratio, while the happening of peeling were impacted by ratio. Here in this study, the response surface methodology with central composite design has been applied, intend to find optimal combinations of pressure and ratio to minimize uniformity and stress, as well as avoid TiN peeling.

Variables:

-- pressure: covariate, quantitative variable, ranges from 4 to 80

-- ratio: covariate, quantitative variable, ranges from 2 to 10

-- stress: quantitative variable, performing as response to be minimized

-- uniformity: quantitative variable, performing as response to be minimized

-- peeling: categorical variable, performing as response to represent the happenness of TiN peeling

Experimental Design

-- Central composite design

The design of experiments follows central composite design, where there're 4 factorial points, 4 star points as well as 3 center points, with $\alpha = \sqrt{2}$, therefore a rotatable design.

Methodology

Quardic Response Surface Model

-- Response surface model is to explore the relationship between several exploratory variables and on or more response variables. Where the main idea is to use a sequence of designed experiments to obtain an optimal response, therefore can be used to optimize a process. While the quardic response surface model is to include both first order of predictors and quardic polynomial terms of predictors into a regression model.

Model Assumptions

-- Similar to regression models, the response surface model assumes normality, independency and constant variance of error term

-- The response surface model assumes that all exploratory varibles are significant

-- A quardic response always has a stationary point, which can be either a min/max or saddle point

Analysis Plan

-- The design of experiments follows the central composite design. From the dataset, we can find that there're 4 factorial points, 4 star points and 3 center point, where the $\alpha = \sqrt{2}$, thus rotatable.

-- Fit two quardic response surface regression models with covariates pressure and ratio, where one it stress response and the other is for uniformity. Visualize the contours for stress and uniformity to find optimial combinations of pressure and ratio to minimize the stress and the uniformity.

-- Add three more data points into the exprimental design, then fit a classification model for peeling status with covariates pressure and ratio, find threshold of the classification model using linear discriminant analysis.

Results

Exploratory analysis

-- Five numbers of summaries for quantitative variables

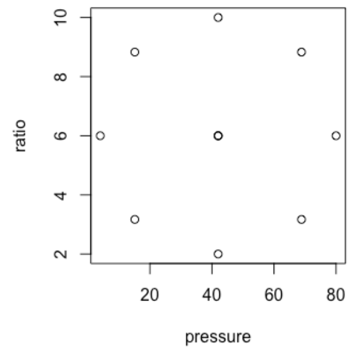
There're 4 quantitative variables interested in the dataset, which are pressure, ratio, stress and uniformity. The five numbers of summaries of them are shown in table1:

table1:

	min	1st Q	median	3rd Q	max
pressure	4	28.57	42	55.44	80
ratio	2	4.585	6	7.415	10
stress	6.49	7.215	7.69	7.97	8.33
uniformity	3.4	5.05	6.2	6.65	8.6

The design of the experimental points is visualized in figure1, where the positions of the points showing a central composite design.

figure1:



To check the setting up of these points, I check the datasets and find that the data does corresponding to the 2 factor central composite design with $\alpha = \sqrt{2}$, where there are 4 factorical points, 4 star points and 3 center points from the dataset.

Statistical analysis

Based on this design, response surface method has been used to fit regression models in terms of the response variables stress and uniformity finding combinations of covariates to minimize the response variables.

-- Model1: response of stress

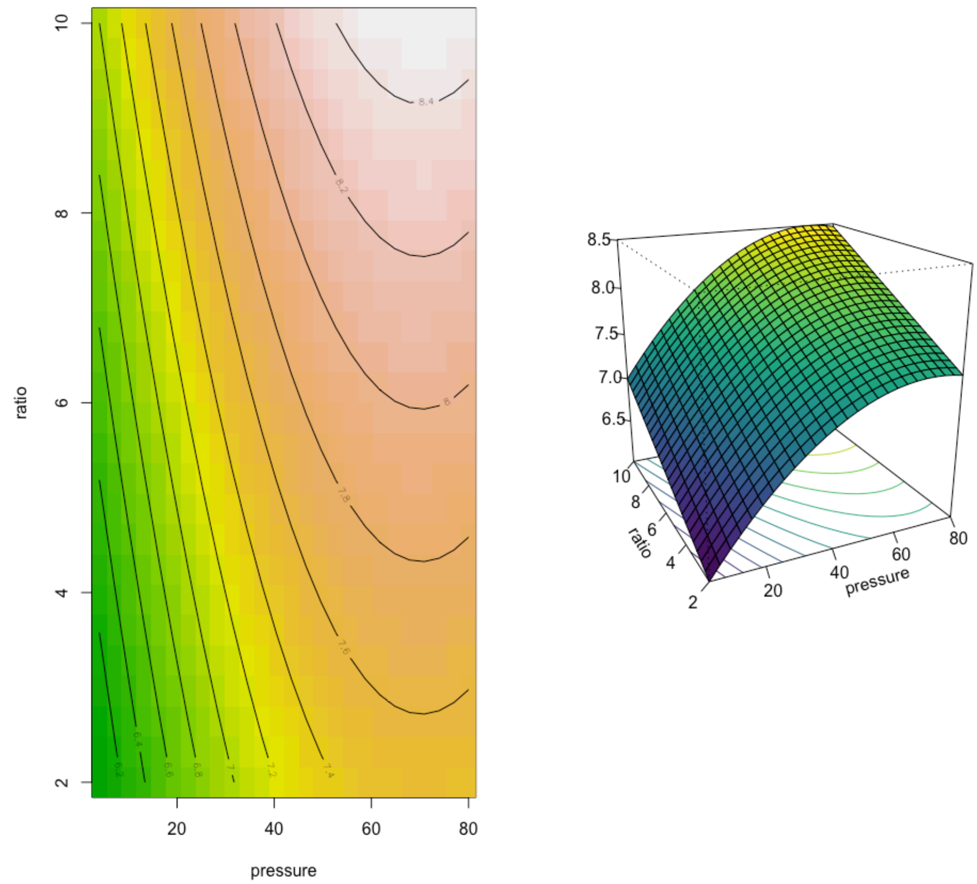
For stress, the second order model has been applied, with first order, interaction as well as polynomial quardic terms of covariates. The outcome has shown that the interaction term and quardic term of ratio were not signifcant, thus an updated models have been tried, which drop either interaction term or quardic term, where the eventual model was the one with first order of pressure and ratio as well as quardic term of pressure . The outcome finally shows signifcancy for all exploratory terms, and the estimated parameters were shown in table2:

table2:

stress	estimate	p-value
intercept	5.5674	$2.319 * 10^{-10}$
pressure	0.0482	$9.843 * 10^{-6}$
ratio	0.1244	$9.55 * 10^{-6}$
pressure^2	-0.0003	0.0002

Based on the model, the stationary point of the response surface was expected to be $\text{pressure} = 70.3256, \text{ratio} = 0$, where such combination is expected to minimize the value of stress. However, based on the ranges of covariates from dataset, the ratio=0 seems to be a little bit out of bound from the ranges of designed experiments, therefore, contours with boundaries corresponding to the covariates from the experimental designed data points were used to visualize the effects from restricted regions. The overall visualizations of stress in terms of pressure and ratio covariates were also shown below in figure2.

figure2:



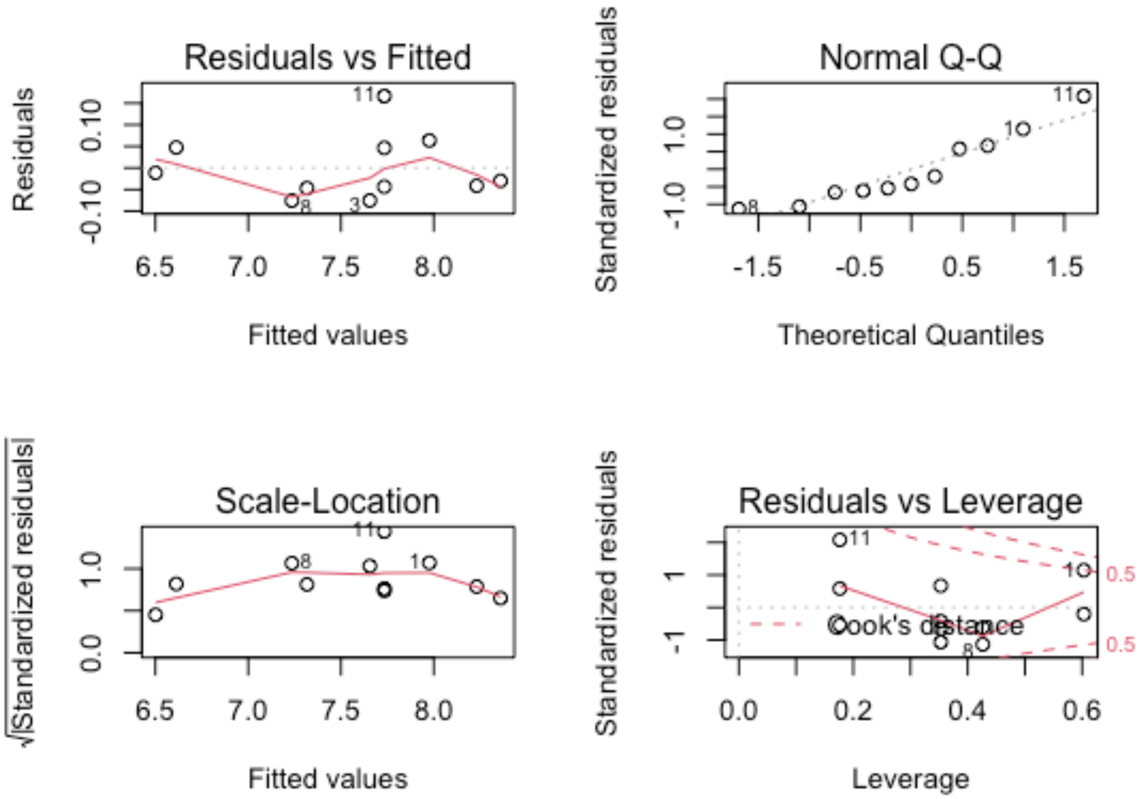
From figure2, it shows a tendency that the minium value of stress can be resulted from smaller value from both pressure and ratio.

-- Model1 fitness and diagnosis

The R-squred value of the model was 0.9849, which was pretty close to 1 and can be seen as a proper model. While in terms of lack of fit test, the p-value was 0.7301, which means do not lack of fit.

To diagnosis the model, residual plots were produced to check if the model meet the error term normalility, independency and constant variance. The plots were shown in figure3:

figure3:



From figure3, we can see that the error term seemed not to be strictly normal distributed, the variance was not constant, also, there're outliers.

-- Model2: response of uniformity

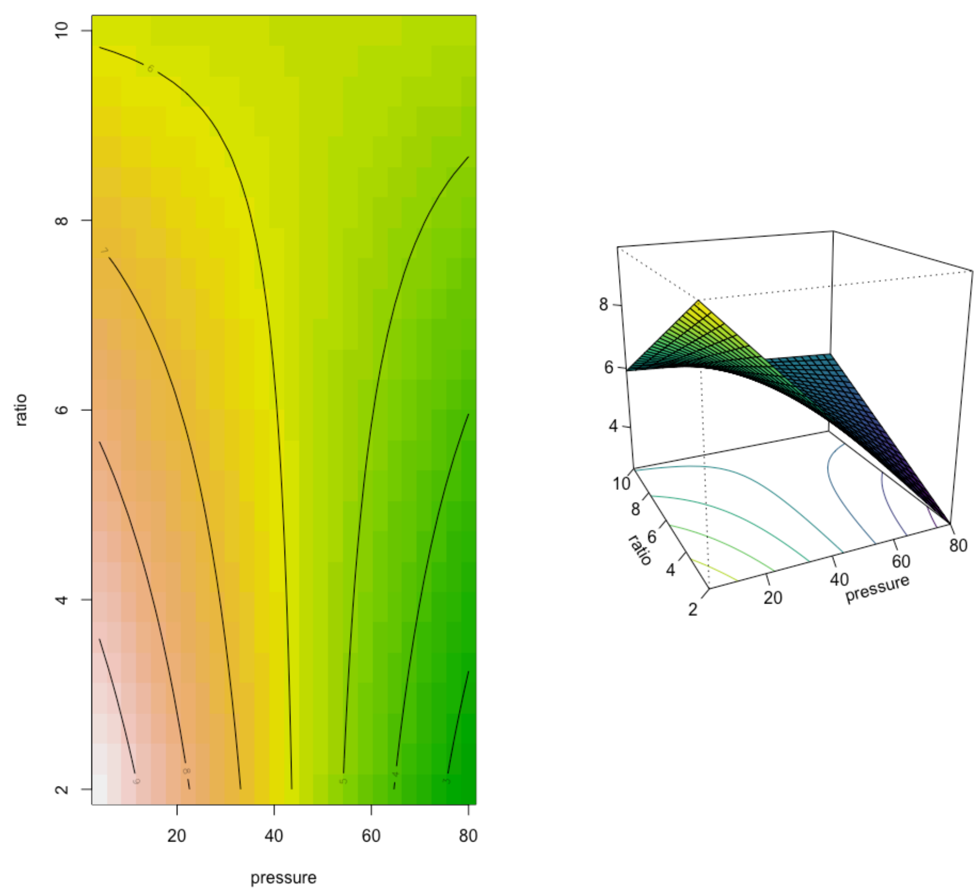
Similarly, another response surface model was used to fit the uniformity from pressure and ratio. The second order model was fitted firstly, however, the outcome showed insignificant interaction and quardic terms of covariates. Therefore, updated models were applied afterwards, where one was with first order of covariates only, another was with first order and interaction term, and the other was with first order and quardic term. By comparing these updated models as well as exploratory variable significancy from the models, the one with both first order of covariates and interaction term performed the best, therefore became the final model. The results of the final model were shown in table3.

table3:

uniformity	estimate	p-value
intercept	11.1952	$3.131 * 10^{-5}$
pressure	-0.1174	0.0025
ratio	-0.5257	0.0260
pressure*ratio	0.0112	0.0282

Besides, a stationary point was suggested from the model, which was pressure=47.0294 and ratio=10.5024, which was expected to minimize the uniformity. However, ratio=10.5024 seems also a little bit out of the bounds of deigned experiments. Contours were then plotted to visualize the uniformity effecting from covariates under the designed ranges, and they were shown in figure4.

figure4:



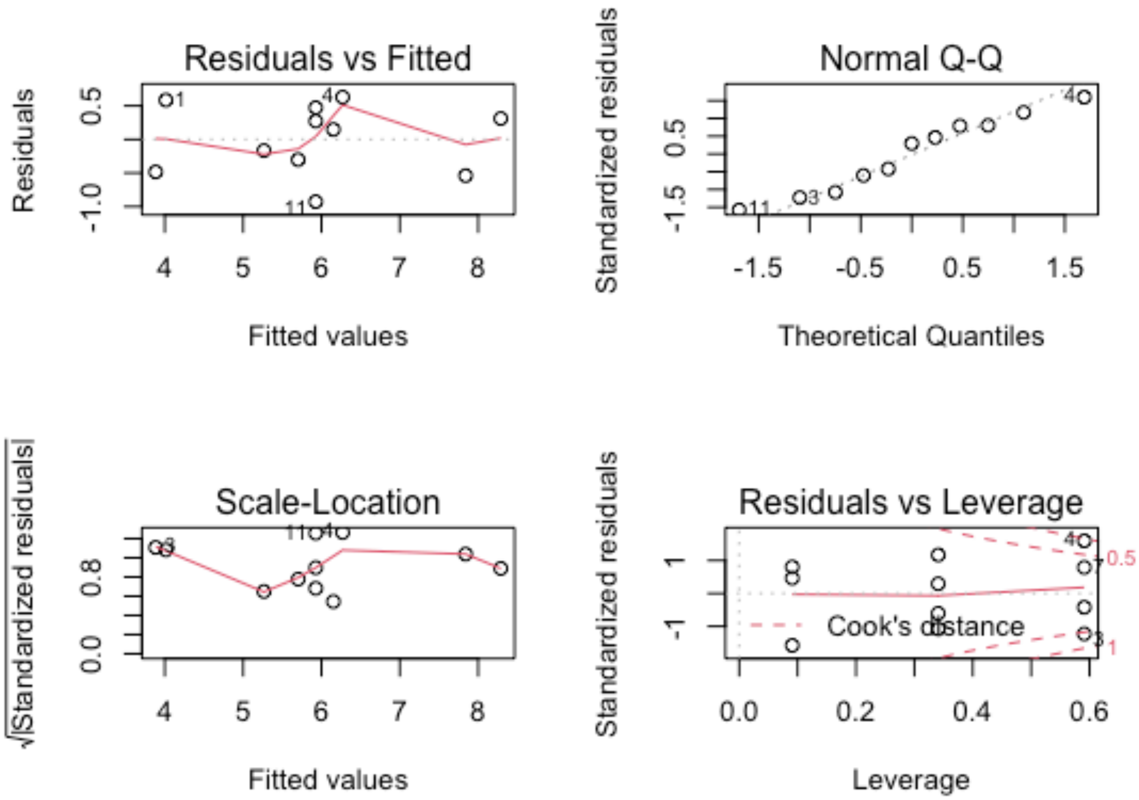
From the plot visualization, it did indicate that an potential minimum can happen at large pressure while small ratio.

-- Model2 fitness and diagnosis

The R-square value of the model was 0.8695, which was pretty close to 1 and can be seen as a proper model. And in terms of lack of fit test, the p-value was 0.7559, therefore dose not lack of fit.

To diagnosis the model, residual plots were produced to check if the model meet the error term normalility, independency and constant variance. The plots were shown in figure5:

figure5:

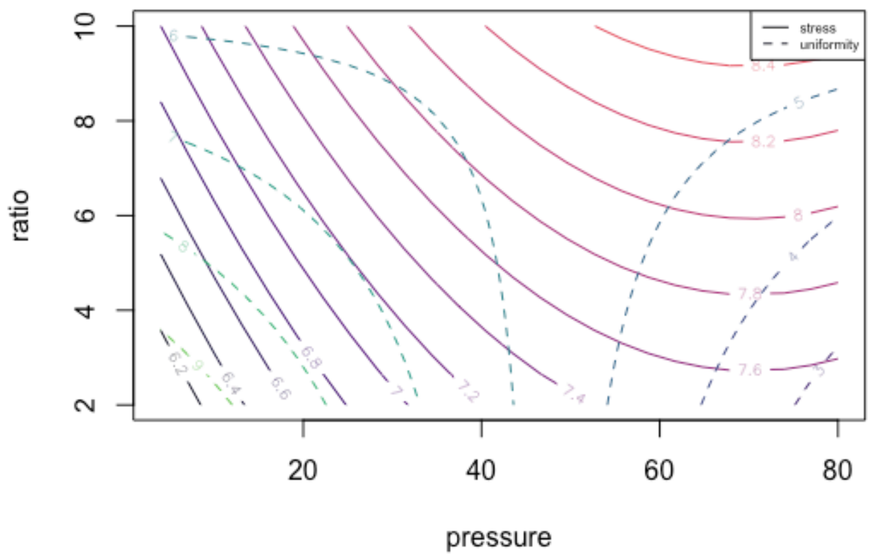


From figure5, we can see that the error term were not normal distributed, the variance was not constant, and outliers also existed.

-- Overlay contours and tradeoffs

Based on figure2 and figure3, it seems to be hard to minimize both stress and uniformity, because the minimizing of stress prefers smaller pressure and ratio, while the minimizing of uniformity prefers larger pressure and smaller ratio. Therefore, it requires some tradeoffs between these two response variables to reach relative optimial status. Contours with levels of these two response variables were then visualize in figure4 for tradeoff exploration.

figure6:

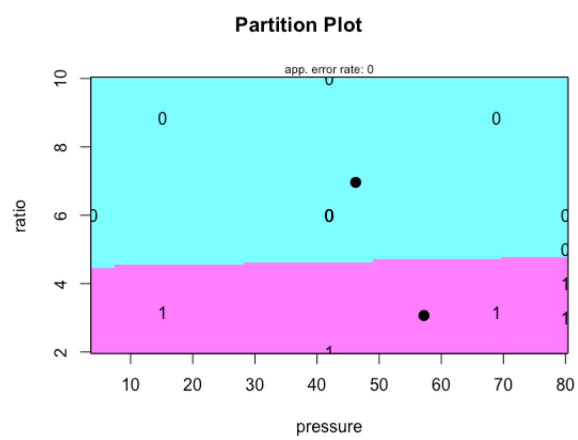


Based in figure6, we can find that the optimial of stress and uniformity both requires smaller ratio, while the tradeoffs would mainly depend on the pressure values.

-- Model3: classification of peeling

For the classification of peeling status, linear discriminant analysis was applied to find a proper threshold to seperate the classes. The results from linear discriminant analysis for peeling was shown in figure7.

figure7:

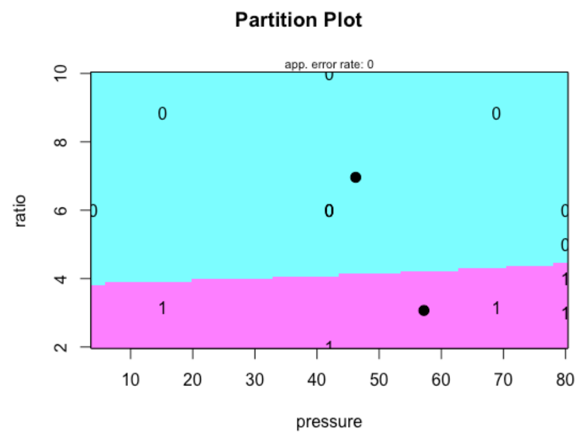


From figure5, we can see that the peeling was pretty relied on the ratio, where the threshold was likely to be ratio=5, where with the ratio value smaller than 5 would be more likely to happen with peeling. To avoid peeling, it's better to have a ratio value greater than 5.

-- Model3 diagnosis

For linear discriminant analysis, the requirement of homogeneity needed to be met. Box's M-test can be used here to test if the covariates were homogeneous. Where the chi-sq test outcome showed a p-value=0.3364, which means the covariates were not significantly homogeneous, the assumption was not met. While quardic discriminant analysis does not require such assumption. By quardic discriminant analysis, the threshold of peeling status was shown in figure8, which also indicated that the peeling was likely to happen at ratio less than 5.

figure8:



Conclusions

Based on the analysis of stress, uniformity and peeling, it is likely that we need a lot tradeoffs among these responses. If we are intended to minimize stress and uniformity and avoid peeling, given that stress was compromised in favor of uniformity, then, a combination of ratio=5 and greater pressure like pressure=80 can potentially be a proper tradeoff.

References

Central Composite Design

Discriminant Analysis

RSM in R

RSM in R

Appendix

```
#### -------------------- Prepare --------------------- ####
setwd('/Users/na/Desktop/MyDoc/MyNIU/2021 Spring/STAT 695/A5')
df <- read.csv('TiN.csv')
head(df)
str(df)

library(rsm)
library(viridis)
```

```
library(MASS)
library(klaR)

#### ---------------------- EDA ---------------------- ####
list(summary(df$pressure), summary(df$ratio),
     summary(df$stress), summary(df$uniformity))
plot(df[,c('pressure', 'ratio')])
(80-42)/(68.87-42)
(10-6)/(8.83-6)

#### -------------- Response Surface Regression ------------- ####
##### stress
m1 <- rsm(stress~SO(pressure, ratio), df)
summary(m1)
m1 <- rsm(stress~FO(pressure, ratio)+TWI(pressure, ratio), df)
summary(m1)
m1 <- rsm(stress~FO(pressure, ratio)+PQ(pressure, ratio), df)
summary(m1)
m1 <- rsm(stress~FO(pressure, ratio)+PQ(pressure), df)
summary(m1)
par(mfrow=c(1,2))
contour(m1, ~pressure+ratio, image = T)
persp(m1, ~pressure+ratio, at=xs(m3), col=viridis(40), contours='colors')
par(mfrow=c(1,1))
car::vif(m1)
par(mfrow=c(2,2))
plot(m1)
par(mfrow=c(1,1))

###### uniformity
m2 <- rsm(uniformity~SO(pressure, ratio), df)
summary(m2)
m2.2 <- rsm(uniformity~FO(pressure, ratio), df)
summary(m2.2)
m2.3 <- rsm(uniformity~FO(pressure, ratio)+TWI(pressure,ratio), df)
summary(m2.3)
m2.4 <- rsm(uniformity~FO(pressure, ratio)+PQ(pressure,ratio), df)
summary(m2.4)
# anova(m2.2, m2.3)
# anova(m2.2, m2.4)
par(mfrow=c(1,2))
contour(m2.3, ~pressure+ratio, image = T)
persp(m2.3, ~pressure+ratio, at=xs(m2.3), col=viridis(40), contours='colors')
par(mfrow=c(1,1))
car::vif(m2.3)
par(mfrow=c(2,2))
```

```
plot(m2.3)
par(mfrow=c(1,1))

#### overlay contours
contour(m1, ~pressure+ratio,at=xs(m1), image = F, col=magma(20), lty=1)
contour(m2.3, ~pressure+ratio, add=T, image = F, col=viridis(10), lty=2)
legend('topright', legend = c('stress', 'uniformity'),
       col = c(magma(20), viridis(10)), lty = c(1,2),
       cex=0.5)

#### adding data points for classification
df2 <- df[,c('pressure', 'ratio', 'peeling')]
df2_add <-matrix(c(80,3,1, 80,4,1, 80,5,0),
                                nrow=3, byrow = T)
colnames(df2_add) <- c('pressure', 'ratio', 'peeling')
df2 <- rbind(df2, df2_add)

## linear discriminant analysis
df2$peeling <- as.factor(df2$peeling)
m3 <- MASS::lda(peeling~pressure+ratio, df2)
klaR::partimat(df2[,c('ratio', 'pressure')], df2$peeling, method='lda')
heplots::boxM(df2[,1:2], df2$peeling)
m3.2 <- MASS::qda(peeling~pressure+ratio, df2)
summary(m3.2)
klaR::partimat(df2[,1:2], df2$peeling, data=df2, method='qda')
```