CHAPTER 2

Work Material Characterization and Elements of Rubber Based Forming Process

Materials characterization is carried out to understand the fundamental properties of a material when subjected to service and environmental loading and operating conditions. Testing helps us to understand and quantify whether a specific material is suitable to a particular application. The characterisation is also very important for FEA simulation and design of any process.

Hence, material characterization of pure copper, Stainless Steel (SS 304) and rubber diaphragms materials viz. natural, nitrile and silicone rubber are carried out to obtain their forming properties. Tensile testing of metals are done to determine yield strength, tensile strength, work hardening exponent and strength coefficient (flow stress at strain value of 1).

The testing of rubber pads are carried out to determine tensile strength and strain of a rubber. Hardness measurement of rubber is also carried out ascertain uniformity in manufacturing of rubbers.

Tensile testing of the pure Copper, SS-304 and rubber sheet samples have been carried out in Universal Testing Machine (UTM).

2.1 Material Properties for Pure Copper Sheet

The tensile test specimen for pure copper, in annealed condition, is shown in figure 2.1. The test has been carried out as per ASTM E8/M standard. The load vs. displacement diagram for the test specimen is shown in figure 2.2. The engineering stress-strain diagram for pure copper sheet is shown in figure 2.3.

The strain tensor can be split into deviatoric and hydrostatic strain tensors.

$$\text{Strain Tensor}(\epsilon) = \epsilon^D + \epsilon^H \tag{2.1}$$

Where,

ϵ^D = Deviatoric strain, strain responsible for plastic deformation

ϵ^H = Hydrostatic strain, responsible for change in the volume of a body, but not its shape.

The total deviatoric strain is equivalent to total plastic strain generated in the component.

The strain shown in the figure 2.3 is the total strains (elastic + plastic strains) in the material. The total strains is decomposed into the elastic and plastic strain components. The plastic strain component is obtained by subtracting the elastic strain , defined as the value of true stress divided by the Young's modulus, from the value of total strain (as shown in figure 2.4) [42]. This can be expressed as

$$\epsilon^{pl} = \epsilon^t - \epsilon^{el} = \epsilon^t - \frac{\sigma}{E} \tag{2.2}$$

where,

ϵ^{pl} is true plastic strain,

ϵ^t is true total strain,

ϵ^{el} is true elastic strain,

σ is true stress, and

E is Young's modulus

The true stress can be expressed in terms of nominal stress and strain as

$$\sigma = \sigma_{nom}(1 + \epsilon_{nom}) \tag{2.3}$$

where, σ_{nom} and ϵ_{nom} are the nominal/ engineering stress and strain.

Table 2.1: Material properties for pure copper sheet

Property	Value
Yield Stress (Y)	78 MPa
Ultimate Tensile Strength (UTS)	239 MPa
Strain Hardening Component (n)	0.44
Strength Coefficient (K)	470
Density	8960 kg/m^3
Young's Modulus	117 GPa
Poisson's Ratio	0.33

The variation of true stress with plastic strain for pure copper is shown in figure 2.5. For the finite element simulation of sheet hydro-forming process, variation of true stress to plastic strain is the input. The material properties for pure copper

Figure 2.1: Tensile test specimen for pure Copper

sheet is shown in table 2.1.

2.1.1 Forming Limit Curve (FLC) for pure copper

In FEM simulation the sheet metal does not fail unless a proper failure criterion is used. FLC developed by Dr. Stuart Keeler in 1960's is the most popularly used failure criterion for sheet metal stamping and for hydro-forming. The key points of Keeler's FLC are:

1. In sheet metal forming, any deformation can be described using Major principal strain and Minor principal strain.

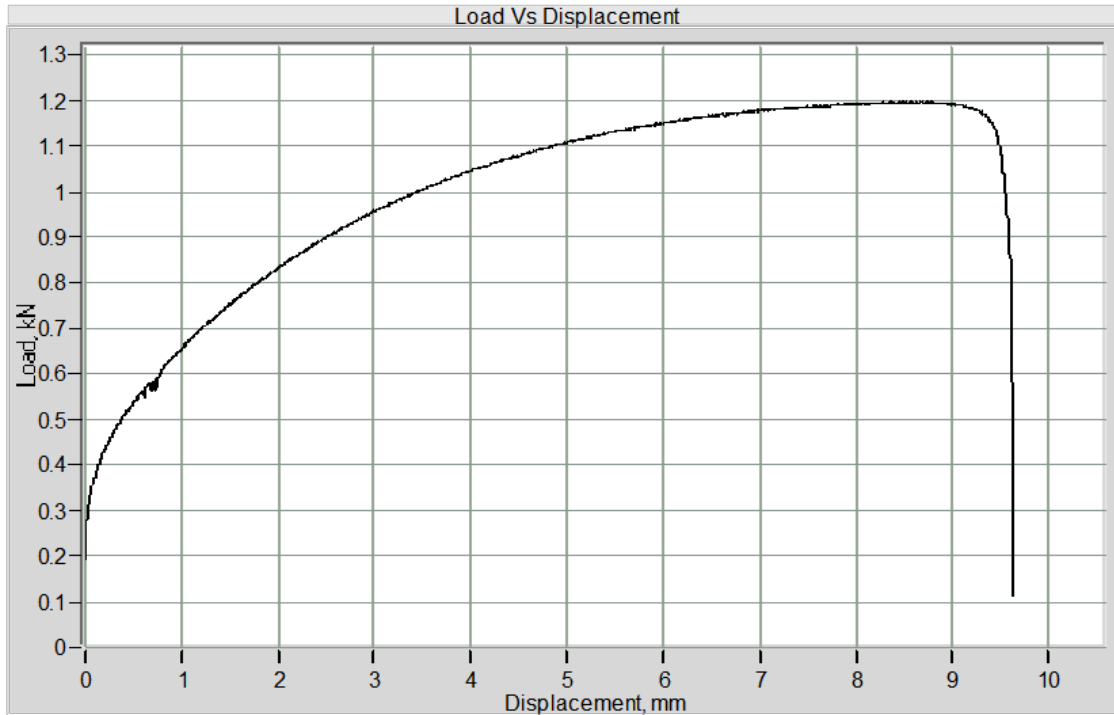2. The shape of the FLC remains constant.

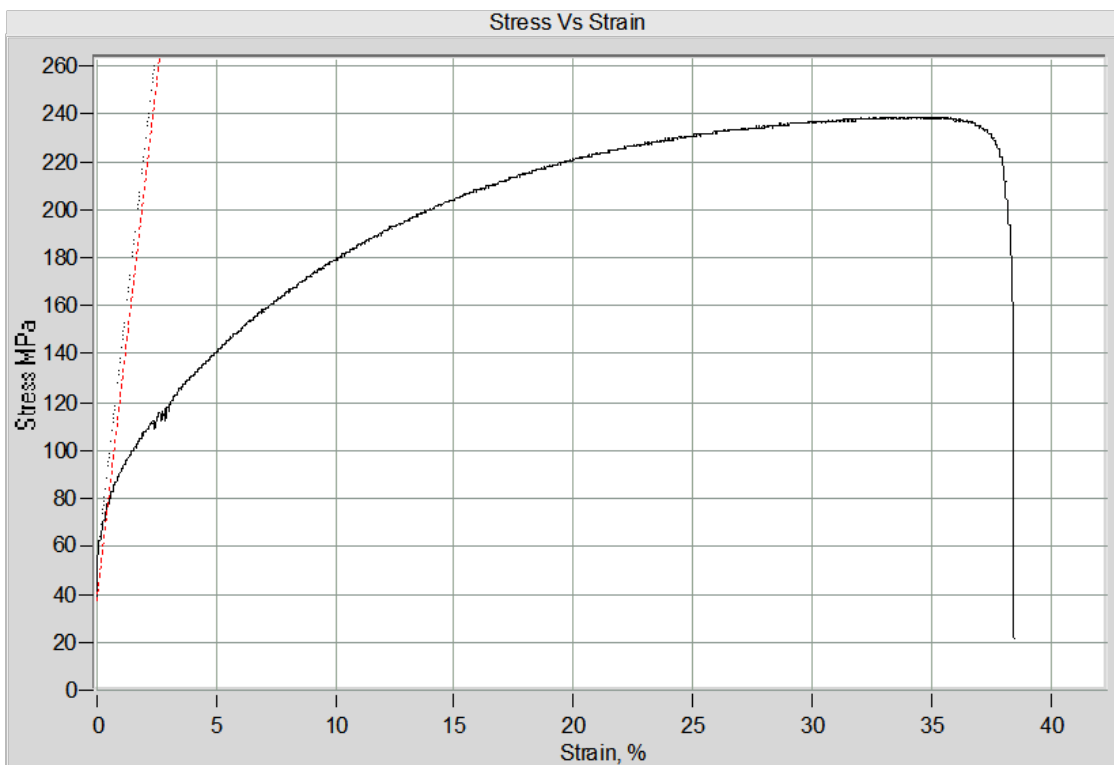Figure 2.2: Load vs. displacement plot for Copper
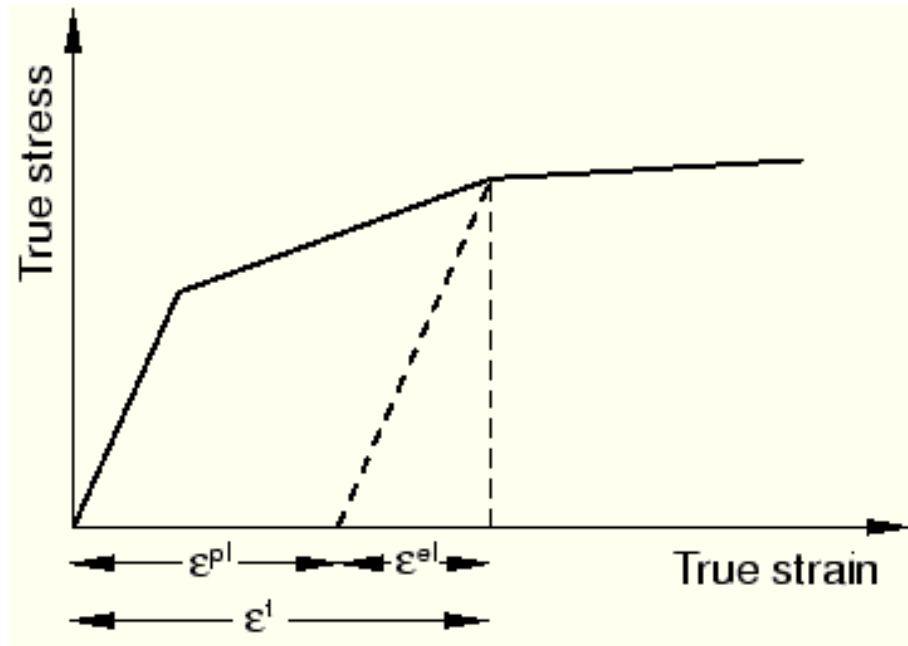

Figure 2.3: Stress-strain diagram of Copper Sheet

Figure 2.4: Decomposition of total strain into elastic and plastic components [42]
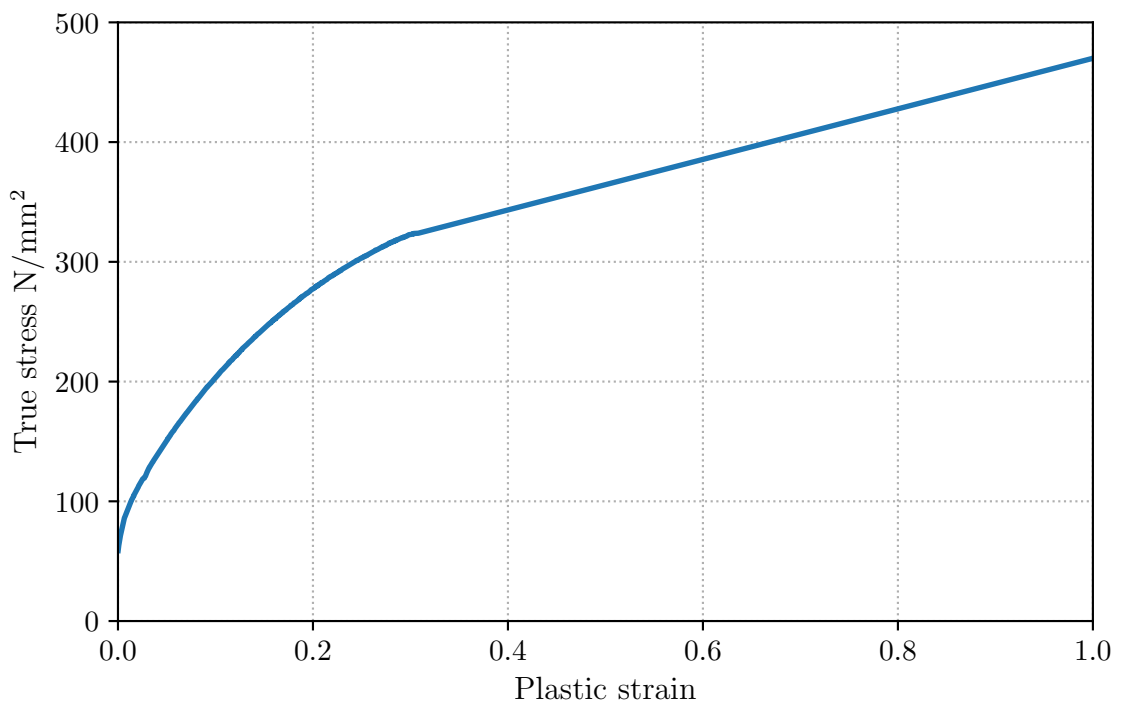
Figure 2.5: True stress vs. Plastic strain for Copper

3. The FLC vertically on a major-minor strain chart based on the position of the
   FLD_0 that is influenced only by the n-value and the thickness of the steel.

4. The marginal curve is generated by subtracting 10% (engineering strain) from
   the FLC.

The FLD_0 in percent engineering strain is defined as:

$$FLD_0 = N.T \tag{2.4}$$

with,

$$N = min(\frac{n}{0.2116}, 1.0)$$

$$T = 23.26 + 14.02min(t, 3.0)$$

where N represents the material effect due to work hardening (n-value) and T represents the thickness effect based on the initial thickness of sheet metal in millimetres.

The shape of the FLC for low-carbon steel can be described with the following equations which are percent engineering strain:

$$e_{maj} = FLD_0 + e_{min}(0.027254e_{min} - 1.1965) \tag{2.5}$$

is for the left side ($e_{min} < 0$) of the curve.

$$e_{maj} = FLD_0 + e_{min}(-0.008565e_{min} + 0.784854) \tag{2.6}$$

is for the right side ($e_{min} > 0$)

Although Keeler developed empirical relations for generating forming limit diagram for steel. The same can be extended for pure copper by using strain hardening coefficient (n) and thickness (t) of copper sheet . The forming limit curve for pure copper based on engineering strains and true strains are shown in figure 2.6 and figure 2.7 respectively.

2.2   Material Properties for Stainless Steel (SS-304)

As discussed in section 2.1, tensile testing is also carried out for samples made out of SS-304 material. Tensile test coupon details are as shown in figure 2.1. The obtained results are presented in table 2.2. For metals, the most commonly used
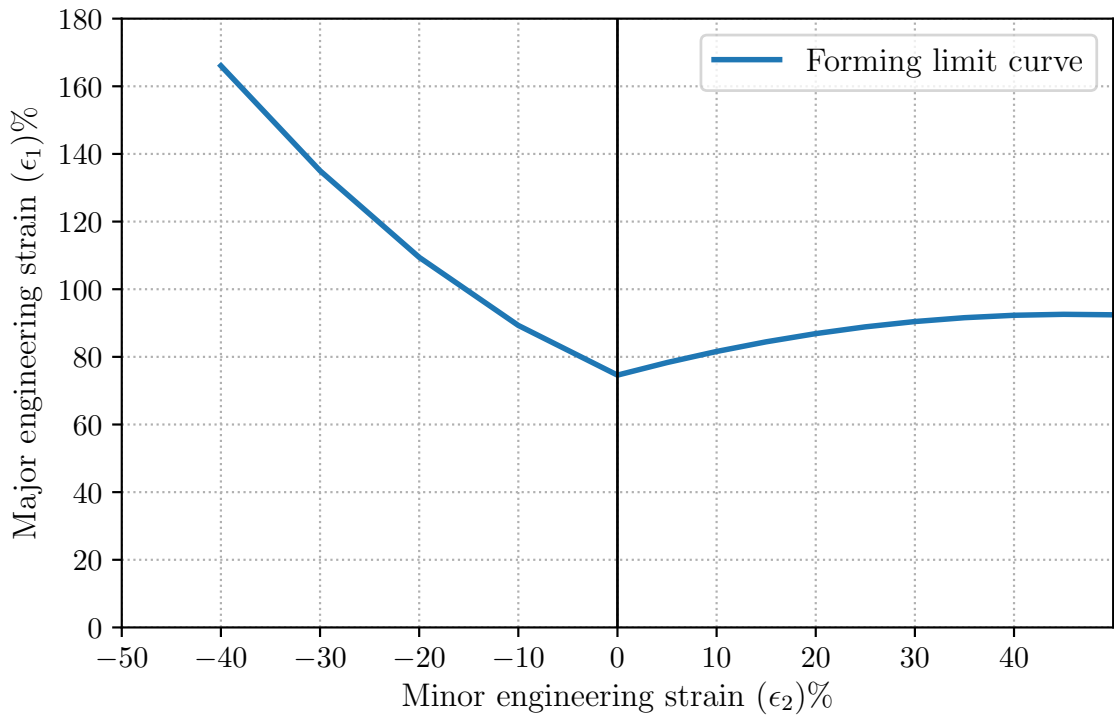
Figure 2.6: Engineering strains based Forming Limit Curve for pure copper
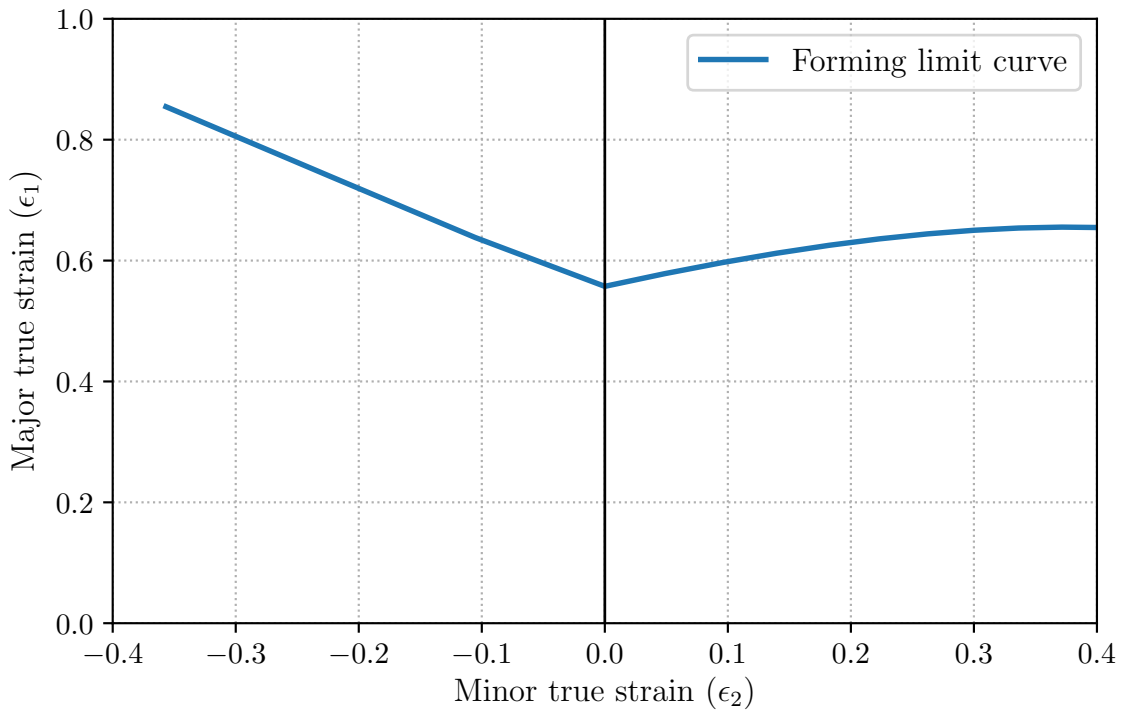

Figure 2.7: True strains based Forming Limit Curve for pure copper

expression for strain hardening is the simple power law [43]. The Power-law is given
by following equation

$$\sigma = K\epsilon^n \tag{2.7}$$

Where, σ = flow stress, K = strength coefficient and n = Strain hardening exponent

The strain hardening index (n) of SS-304 is 0.4 which explains that the material
is very much suitable for forming application. The strength coefficient (K) is 1540
MPa. The plastic strain and the corresponding flow stress is presented in figure 2.8.

Table 2.2: Material Properties for Stainless Steel (SS-304)

Property	Value
Yield stress(Y)	297.54 MPa
Ultimate Tensile strength(UTS)	715.69 MPa
Strain Hardening index(n)	0.4
Strength Coefficient(K)	1540.348
Density	7800 kg/m^3
Young's Modulus(E)	210 GPa
Poisson's Ratio	0.3

2.3   Material Properties for Rubber

Rubber, elastic substance obtained from the exudations of certain tropical plants
(natural rubber) or derived from petroleum and natural gas (synthetic rubber).
Because of its elasticity, resilience, and toughness, rubber is the basic constituent
of the tires used in automotive vehicles, aircraft, and bicycles. The main chemical
constituents of rubber are elastomers, or "elastic polymers", large chainlike molecules
that can be stretched to great lengths and yet recover their original shape.
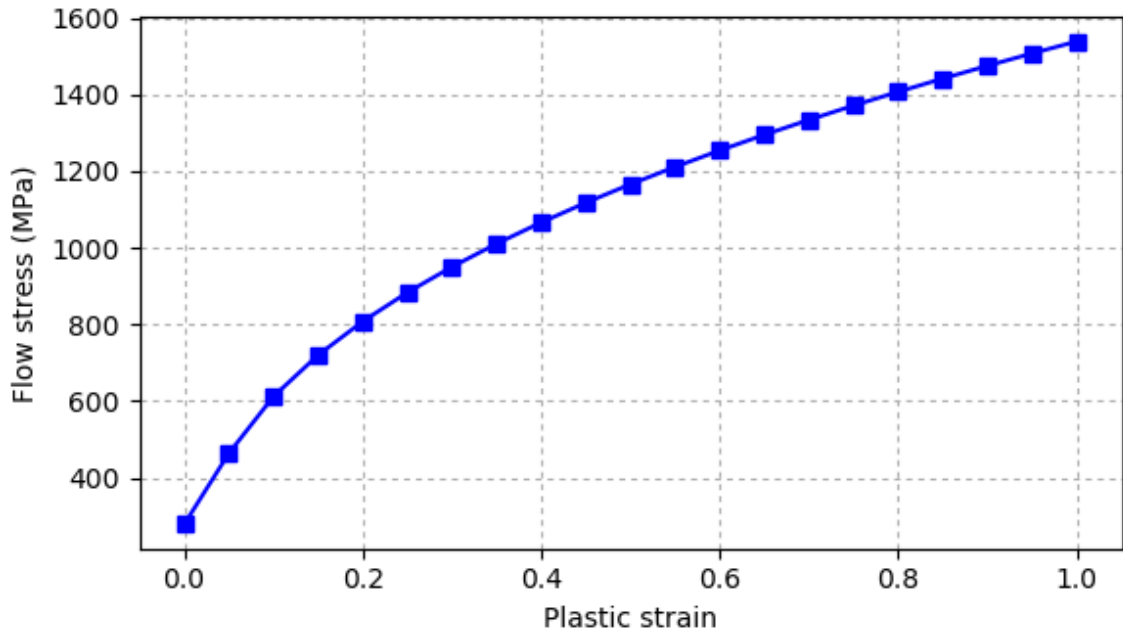
Figure 2.8: Plastic stress vs. Flow stress for SS-304

The rubbers can be classified into five categories based on its constituent. The classification is shown in figure 2.9.
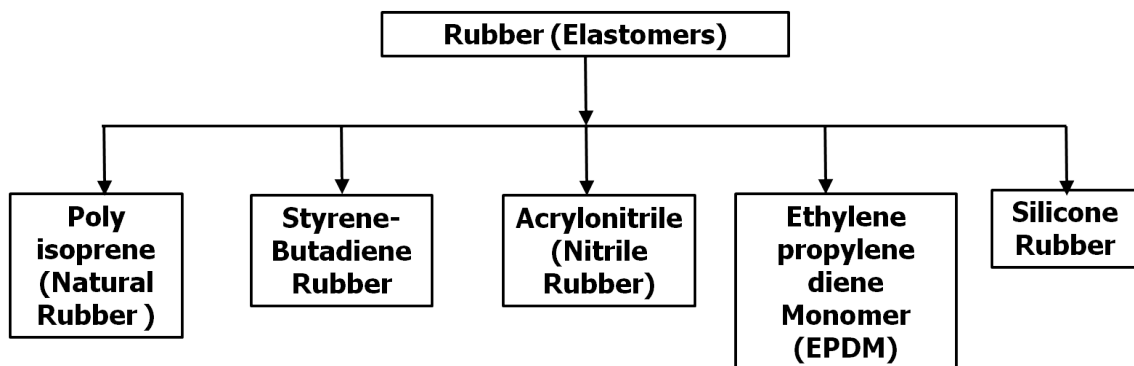

Figure 2.9: Rubber classification

The first common elastomer was polyisoprene, from which natural rubber is made. Natural rubber continues to hold an important place in the market today; its resistance to heat buildup makes it valuable for tires used on racing cars, trucks, buses, and airplanes. Nevertheless, it constitutes less than half of the rubber produced commercially; the rest is rubber produced synthetically by means of chemical processes that were partly known in the 19th century but were not applied commercially until the second half of the 20th century, after World War II. Among the most important synthetic rubbers are butadiene rubber, styrene-butadiene rubber, neo-

prene, the butyl rubber, and the silicones. Synthetic rubbers, like natural rubbers, can be toughened by vulcanization and improved and modified for special purposes by reinforcement with other materials.

In the present work natural rubber, B-Nitrile rubber and Silicon rubber have been evaluated for their suitability in forming applications.

2.3.1   Steps in Rubber Manufacturing

The properties of rubber can be altered by modifying its constituents. For instance, natural rubber can be produced to different level of shore hardness by altering filler elements and some of its constituents. In present work, natural rubber having harness of 50 A and 60 A has been manufactured. Each new type of rubber exhibits different level of hyper-elasticity and resistance to stretching. This flexibility of developing new rubbers can add lot of research aspects in the field of RBSH process [44].

Rubber processing consists of four basic steps: (1) mastication, when the elastomer is sheared and the molecules are broken down to give easier flow, (2) mixing, usually carried out immediately after mastication, when additives are incorporated, (3) shaping of the viscous mass, for example,by extrusion or molding, and (4) curing, when the polymer molecules become interlinked and the shape is fixed.

Mastication

Mastication and softening are usually carried out in batches. The operation is done either in large enclosed mixing machines or on rubber mills. The preeminent example of an enclosed machine is the Banbury (registered trademark) mixer, consisting of heavy steel counterrotating paddles in an hourglass-shaped chamber, holding up to one-half ton of rubber. Rubber mills have two large horizontally opposed, closely spaced steel cylinders, up to 3 metres (10 feet) long, that are rotated slowly in opposite directions and at somewhat different speeds. Rubber is sheared and softened in the gap between the paddles and wall of the Banbury mixer and in the gap between the two cylinders in the roll mill.

Mixing

Mixing is carried out on machines similar to those used in mastication, sometimes immediately after softening. Reactive materials, fillers, oils, and protective chemicals of various kinds, as described above, are incorporated into the base elastomer by a combined shearing and mixing action. An enclosed Banbury-type mixer can produce up to one-half ton of mixed compound in a few minutes. The compound is then sheeted out, coated with a release soap to prevent sticking, and stored until use on steel pallets that can hold up to one ton of rubber.

Shaping

Shaping of the mixture into the desired form takes place in several ways. Extruders are used to produce long continuous products such as tubing, tire treads, and wire coverings. They are also used to produce various profiles that can later be cut to length. Multiroll calenders are used to make wide sheeting. In transfer and injection molds, the rubber mix is forced through channels into a mold chamber of the required shape, where it is cured under pressure. Tires are made of several components: bead wire, sidewall compound, inner liner, cord plies, belt package, and tread; these are brought together and assembled as a complete tire before being transferred to the curing press.

Curing

Curing is carried out in pressurized steel molds, which are heated by steam or electricity to temperatures at which the interlinking reaction takes place. Typical

cure conditions are several minutes at a temperature of 160 ^{o}C (320 ^{o}F). Because
heat penetrates rubber slowly, thick articles must be allowed longer curing times, up
to several hours, at lower temperatures. Pressures of 1megapascal (145 pounds per
square inch) or more are normally imposed in order to maintain the desired shape
and to force trapped air to dissolve in the compound. Other methods of curing the
rubber mix after it has been shaped include steam heating in autoclaves, microwave
irradiation,and passage through a heated bath of molten metal salts or a fluidized
bed. In these cases curing is carried out at near-atmospheric pressure.
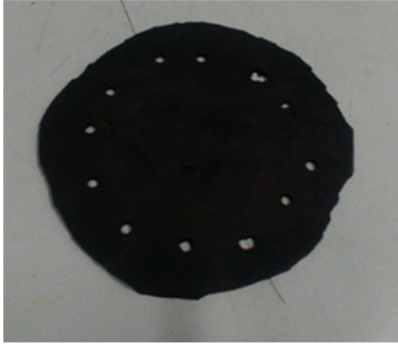
2.3.2 Material Characterisation

Characterisation of rubber is first thing to be carried out before to be used as
rubber diaphragm. Geiger and Sprenger [28] has conducted a study on the character-
ization of polyurethane pads and experimental bending using elastomer pads. Most
of the application uses polyurethane as Rubber Material for operations because of
its elastic nature and long life. However, Natural Rubber, silicon rubber, B-nitrile
rubbers can also be a potential candidate for deep drawing operations. Natural
rubber derived from the tree Heveabrasiliensis is the prototype of a wide range of
materials which have a high extensibility combined with an ability to recover from
extension. It is usual to refer to these materials as highly elastic, and to group their
properties as high elasticity.

Natural rubber exhibits high tensile strength and elastic property at room tem-
perature. The major constituent of this natural Rubber is Cis1,4 Polyisoprene.
The uniqueness of natural rubber lies in its physical properties of extensibility and
toughness, i.e., its ability to be stretched repeatedly to seven or eight times than
its original length. In the absence of tensile (stretching) stress, the polymer chains
assume an amorphous, or disordered, arrangement. On being stretched, however,
the molecules readily align into an ordered crystalline arrangement. Crystallinity
lends greater strength to the material therefore natural rubber is considered to be
"self-reinforcing" [45].

Silicone rubber is the best among all the elastomers for both high and low tem-
perature. It has excellent ozone, weather resistance, and electrical insulation. Me-

SI.No.	Ingredients	PHR	Actual Weighed	Property/composition
1	ISNR5,	80×23	1840	Rubber
2	SBR 1502	20	460	Rubber
3	TMQ	1	23	Anti-oxidant
4	6PPD	2	46	Anti-oxidant
5	Zinc	5	115	Activators for curing
6	Oxide,	2	46	Hardness, strength,
7	Stearic	3	69	softness
8	acid	36	828	
9	CI Resin	7	161	Curing chemicals
	HAF Black			
10	Naphthenic	1.2	27.6	
11	oil	1.5	34.5	
12	MBTS,	1	23	
	TMTD,			
	Sulphur			

Chemical Composition of Natural Rubber	Natural rubber

Figure 2.10: Composition of Natural Rubber

chanical properties such as tensile strength, are low but changes very little when measured at higher temperatures. Applications include aerospace, medical, food contact, and automotive ignition cable [46]. B-nitrile rubber is the workhorse of the marketplace for its oil resistant properties. This rubber is known as acrylonitrile butadiene, Buna-N & simply nitrile according to the chemist, to other & Industrial firm respectively. It is a better heat aging resistance. Special compounding ingredients can be added to increase heat aging resistance. Like SBR, NBR needs reinforcing fillers to give good mechanical properties.

Natural Rubber

The composition of natural rubber is shown in figure 2.10. The stress-strain diagram for rubber sheet is shown in figure 2.11. The material properties for rubber sheet is shown in table 2.3.

Table 2.3: Material properties of Natural Rubber

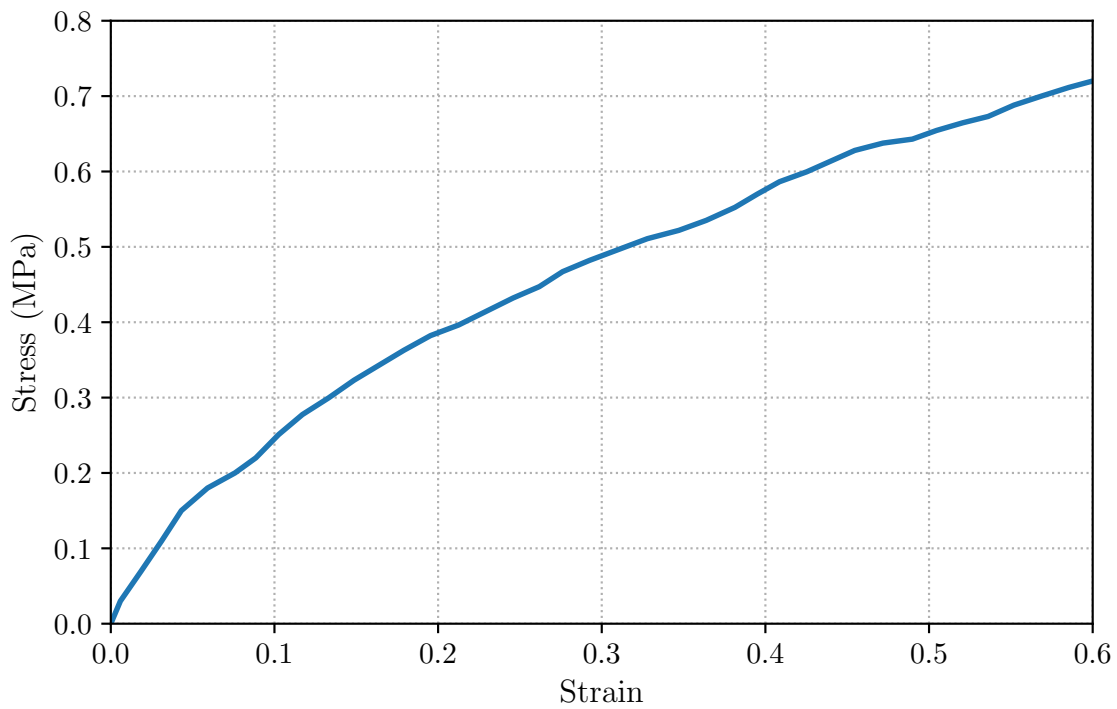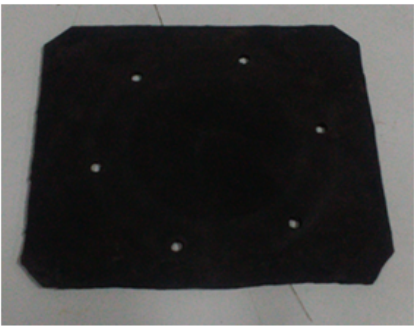| Property	Value
Size	ϕ 100× 4 mm
Density	910 kg/m^3
Young's Modulus	0.001 GPa
Poisson's Ratio	0.4997

Figure 2.11: Stress-Strain diagram for Natural Rubber

Nitrile Rubber

The composition of Nitrile Rubber is shown in figure 2.12. The material properties such as shore hardness, ultimate strength and percentage elongation are given in table 2.4.

SI.No.	Ingredients	PHR	Actual Weighed	Property/composition
1	Krynac 2645F	100×36	3600	Nitrile raw rubber Anti-oxidants
2	6PPD	1.5	54	
3	TMQ	1	36	Activators for curing
4	Sulphur	0.8	28.8	
5	Zinc	5	180	Hardness, strength,
6	Oxide	1	36	elongation
7	Stearic	20	720	Curing chemicals
8	acid	2	72	
9	SRF Black	3	108	
10	DBP(oil)	1	36	
11	CI Resin MBTS, TMTD	1.2	43.2	
Chemical Composition of Nitrile Rubber				Nitrile rubber

Figure 2.12: Composition of Nitrile Rubber

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Table 2.4: Comparative material properties for Rubber

S.No.	Properties	Natural Rubber	Silicon Rubber	Nitrile Rubber
1	Thickness (mm)	3	3	3
2	Hardness(Shore-A)	61 62	57 60	57.5 57.8
3	UTS (MPa)	13.07	6.75	9.25
4	Elongation at break(%)	378	209	358

Silicone Rubber

The composition of Silicone Rubber is shown in figure 2.13. The material properties such as shore hardness, ultimate strength and percentage elongation are given in table 2.4.

Figure 2.13: Composition of Silicone Rubber

Comparative Analysis of Rubber

In order to maintain the uniformity, rubber diaphragm of same thickness i.e 3 mm are manufactured. The constituents are selected in such a way to achieve Shore A hardness in the range of 55-60. Table 2.4 lists out the mechanical properties of all three rubbers.

The comparative load displacement curve and stress-strain curve are shown in figure 2.14 and figure 2.15 respectively. It is evident from the comparative stress-strain plot that natural rubber exhibits maximum tensile strength of 13.07 MPa and percentage elongation of 378%. Hence, Natural rubber is most suitable for forming

application where strength and elongation requirement are on higher side. Silicone rubber exhibits maximum tensile strength of 6.75 MPa, but Young's Modulus is more than Natural rubber. Thus, Silicone rubber would exert minimum resistance during forming. Properties of Nitrile rubber lies between Natural rubber and Silicone rubber. It exhibits maximum tensile strength of 9 MPa and percentage elongation of 350%.
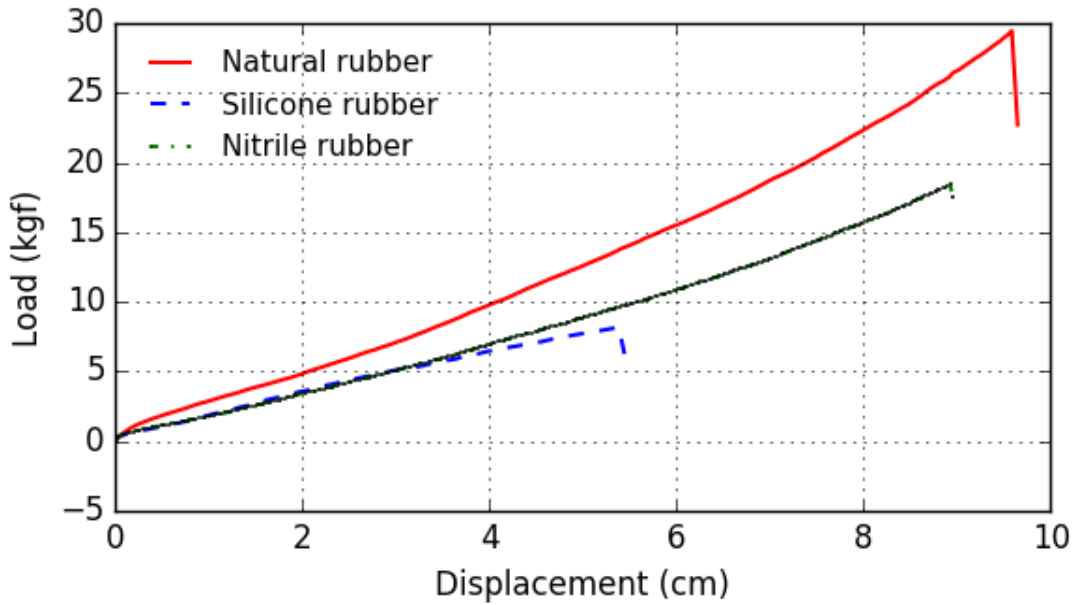

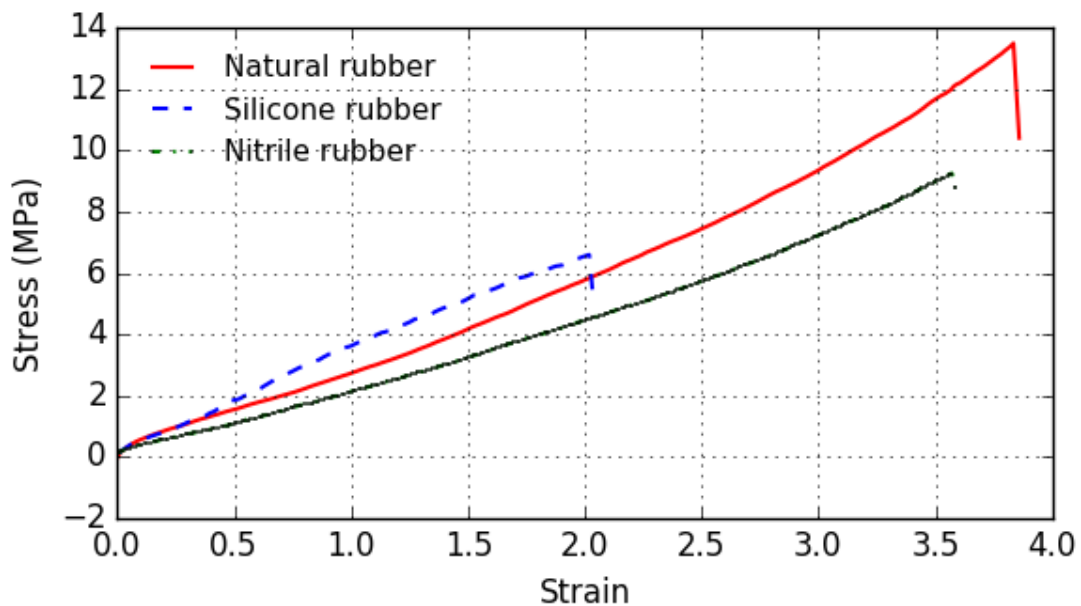Figure 2.14: Comparison of load displacement curve for three rubbers

Figure 2.15: Comparison stress-strain curves for three rubbers

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